PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of
ASTLES, et al.

Examiner: CHANG, Celia

Group Art Unit.: 1625

Application No.: **09/843,126**

Filed: **04/26/2001**

Title: **CHEMICAL COMPOUNDS**

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Dear Sir:

Applicants submit herewith certified copy of GB0012362.0 application, filed on May 22, 2000, for which priority is claimed in the above-identified application.

This submission and request for entry is being made to satisfy the requirements under 35 U.S.C. § 119. Please note that no fees are associated with the entry of the priority documents since they are being timely submitted prior to the date the issue fee is due.

Respectfully submitted,

June 25, 2003

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Aventis Docket No. USCA2413 US NP

Patents Form 1/77

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Continuation sheets of this form	
Description	122
Claim(s)	
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10. If you are also filing any of the following, state how many against each item.

Priority documents	
Translations of priority documents	
Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	
Request for preliminary examination and search *(Patents Form 9/77)*	
Request for substantive examination *(Patents Form 10/77)*	
Any other documents *(please specify)*	

11. I/We request the grant of a patent on the basis of this application.

Signature Lee Caffin LEE CAFFIN Date 22/05/2000

Agent for the Applicant

12. Name and daytime telephone number of person to contact in the United Kingdom LEE CAFFIN 0181 919 2722

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1. Your reference — CA2413

2. Patent application number *(The Patent Office will fill in this part)* — **0012362.0**

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

AVENTIS PHARMA LIMITED
AVENTIS HOUSE
50 KINGS HILL AVENUE
KINGS HILL
WEST MALLING
KENT ME19 4AH

Patents ADP number *(if you know it)*

If the applicant is a corporate body, give the country/state of its incorporation — GB

4. Title of the invention — CHEMICAL COMPOUNDS

5. Name of your agent *(if you have one)* — LEE CAFFIN

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

AVENTIS PHARMA LIMITED
RAINHAM ROAD SOUTH
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ESSEX RM10 7XS

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6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

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Number of earlier application	Date of filing *(day / month / year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*

a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.
See note (d))

YES

INVESTOR IN PEOPLE

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NP10 8QQ

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I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears an amendment, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

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Dated 27 May 2003

CHEMICAL COMPOUNDS

This invention is directed to substituted benzylamines, their preparation, pharmaceutical compositions containing these compounds, and their pharmaceutical use in the treatment of disease states capable of being modulated by the inhibition of tryptase.

Tryptase is stored in mast cell secretory granules and is the major secretory protease of human mast cells. Tryptase has been implicated in a variety of biological processes, including degradation of vasodilating and bronchorelaxing neuropeptides (Caughey, et al., J. Pharmacol. Exp. Ther., 1988, 244, pages 133-137; Franconi, et al., J. Pharmacol. Exp. Ther., 1988, 248, pages 947-951; and Tam, et al., Am. J. Respir. Cell Mol. Biol., 1990, 3, pages 27-32) and modulation of bronchial responsiveness to histamine (Sekizawa, et al., J. Clin. Invest., 1989, 83, pages 175-179). As a result, tryptase inhibitors may be useful as anti-inflammatory agents (K Rice, P.A. Sprengler, Current Opinion in Drug Discovery and Development, 1999, 2(5), pages 463-474) particularly in the treatment of chronic asthma (M.Q. Zhang, H. Timmerman, Mediators Inflamm., 1997, 112, pages 311-317) and may also be useful in treating or preventing allergic rhinitis (S. J. Wilson et al, Clin. Exp. Allergy, 1998, 28, pages 220-227), inflammatory bowel disease (S.C. Bischoff et al, Histopathology, 1996, 28, pages 1-13), psoriasis (A. Naukkarinen et al, Arch. Dermatol. Res., 1993, 285, pages 341-346), conjunctivitis (A.A.Irani et al, J. Allergy Clin. Immunol., 1990, 86, pages 34-40), atopic dermatitis (A. Jarvikallio et al, Br. J. Dermatol., 1997, 136, pages 871-877), rheumatoid arthritis (L.C Tetlow et al, Ann. Rheum. Dis., 1998, 54, pages 549-555), osteoarthritis (M.G. Buckley et al, J. Pathol., 1998, 186, pages 67-74), gouty arthritis, rheumatoid spondylitis, and diseases of joint cartilage destruction.

In addition, tryptase has been shown to be a potent mitogen for fibroblasts, suggesting its involvement in the pulmonary fibrosis in asthma and interstitial lung diseases (Ruoss et al., J. Clin. Invest., 1991, 88, pages 493-499). Therefore, tryptase inhibitors may be useful in treating or preventing fibrotic conditions (J.A. Cairns and A.F. Walls, J. Clin. Invest., 1997, 99, pages 1313-1321) for example, fibrosis, sceleroderma, pulmonary fibrosis, liver cirrhosis, myocardial fibrosis, neurofibromas and hypertrophic scars.

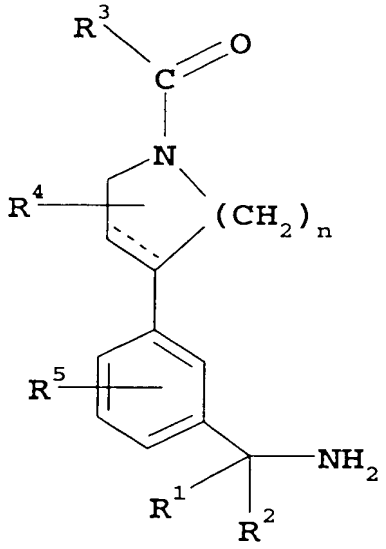
Additionally, tryptase inhibitors may be useful in treating or preventing myocardial infarction, stroke, angina and other consequences of atherosclerotic plaque rupture (M. Jeziorska et al, J. Pathol., 1997, 182, pages 115-122). Tryptase has also been discovered to activate prostromelysin which in turn activates collagenase, thereby initiating the destruction of cartilage and periodontal connective tissue, respectively. Therefore, tryptase inhibitors could be useful in the treatment or prevention of arthritis, periodontal disease, diabetic retinopathy, and tumor growth (W.J. Beil et al, Exp. Hematol., (1998) 26, pages 158-169). Also, tryptase inhibitors may be useful

in the treatment of anaphylaxis (L.B. Schwarz et al, J. Clin. Invest., 1995, 96, pages 2702-2710), multiple sclerosis (M. Steinhoff et al, Nat. Med. (N. Y.), 2000, 6(2), pages 151-158), peptic ulcers and syncytial viral infections.

Mast cell mediated inflammatory conditions, in particular asthma, are a growing public health concern. Asthma is frequently characterized by progressive development of hyper-responsiveness of the trachea and bronchi to both immunospecific allergens and generalized chemical or physical stimuli, which lead to the onset of chronic inflammation. Leukocytes containing IgE receptors, notably mast cells and basophils, are present in the epithelium and underlying smooth muscle tissues of bronchi. These leukocytes initially become activated by binding of specific inhaled antigens to the IgE receptors and then release a number of chemical mediators. For example, degranulation of mast cells leads to the release of proteoglycans, peroxidase, arylsulfatase B, tryptase and chymase, which results in bronchiole constriction.

We have now found a novel group of substituted benzylamines which have valuable pharmaceutical properties, in particular the ability to inhibit tryptase.

Thus, in one aspect, the present invention is directed to compounds of general formula (I):-



(I)

wherein:-

------ is a single or a double bond;

R^1 and R^2 are each independently hydrogen or lower alkyl;

R^3 is aryl, arylalkenyl, cycloalkenyl, cycloalkyl, heteroaryl, heteroarylalkenyl, heterocycloalkenyl, a carbon linked heterocycloalkyl or alkyl optionally substituted by one or more groups selected from hydroxy, alkoxy, alkyloxycarbonylamino, cycloalkyl, heterocycloalkyl, R^6, $-OR^6$, $-S(O)_mR^6$ or $-C(=O)-R^6$;

R^4 is hydrogen, acyl, alkoxy, alkyloxycarbonyl, carboxy, cyano, halo, hydroxy, $-C(=O)-NY^1Y^2$ or alkyl optionally substituted with alkoxy, alkylcarbonylamino, alkylsulfonylamino, hydroxy, $-S(O)_m$-alkyl or $-NY^1Y^2$;

R^5 is hydrogen, acyl, alkoxy, alkyloxycarbonyl, aryl, carboxy, cyano, halo, heteroaryl, heteroaryloxy, heterocycloalkyl, heterocycloalkyloxy, heterocycloalkylalkyloxy, heteroarylalkyloxy, hydroxy, trifluoromethyl, $-C(=O)-NY^1Y^2$, $-NY^1Y^2$, $-Z^1-C_{2-6}$alkylene-R^7 or alkyl optionally substituted with alkoxy, alkylcarbonylamino, alkylsulfonylamino, aryl, heteroaryl, heterocycloalkyl, hydroxy, ureido, $-C(=O)-NY^1Y^2$, $-SO_2-NY^1Y^2$, $-S(O)_m$-alkyl or $-NY^1Y^2$;

R^6 is aryl or heteroaryl;

R^7 is hydroxy, alkoxy, ureido, $-C(=O)-NY^1Y^2$, $-SO_2-NY^1Y^2$, $-S(O)_m$-alkyl or $-NY^1Y^2$;

R^8 is hydrogen or lower alkyl;

Y^1 and Y^2 are independently hydrogen, alkenyl, alkyl, aryl, arylalkyl, cycloalkyl, heteroaryl, heteroarylalkyl or heterocycloalkyl; or the group $-NY^1Y^2$ may form a cyclic amine;

Z^1 is O, $S(O)_m$ or NR^8;

m is zero or an integer 1 to 2;

n is zero or an integer 1 to 4;

and the corresponding N-oxides, and their prodrugs; and pharmaceutically acceptable salts and solvates (e.g. hydrates) of such compounds and their N-oxides and prodrugs.

In the present specification, the term "compounds of the invention", and equivalent expressions, are meant to embrace compounds of general formula (I) as hereinbefore described, which expression includes the prodrugs, the pharmaceutically acceptable salts and the solvates, e.g. hydrates, where the context so permits. Similarly, reference to intermediates, whether or not they themselves are claimed, is meant to embrace their salts, and solvates, where the context so permits. For the sake of clarity, particular instances when the context so permits are sometimes indicated in the text, but these instances are purely illustrative and it is not intended to exclude other instances when the context so permits.

As used above, and throughout the description of the invention, the following terms, unless otherwise indicated, shall be understood to have the following meanings:-

"Patient" includes both human and other mammals.

"Effective amount" is meant to describe an amount of compound of the present invention effective in inhibiting tryptase and thus producing the desired therapeutic effect.

"Acyl" means an H-CO- or alkyl-CO- group in which the alkyl group is as described herein.

"Acylamino" is an acyl-NH- group wherein acyl is as defined herein.

"Alkenyl" means an aliphatic hydrocarbon group containing a carbon-carbon double bond and which may be straight or branched having about 2 to about 15 carbon atoms in the chain. Preferred alkenyl groups have 2 to about 12 carbon atoms in the chain; and more preferably about 2 to about 4 carbon atoms in the chain. "Branched", as used herein and throughout the text, means that one or more lower alkyl groups such as methyl, ethyl or propyl are attached to a linear chain; here a linear alkenyl chain. "Lower alkenyl" means about 2 to about 4 carbon atoms in the chain which may be straight or branched. Exemplary alkenyl groups include ethenyl, propenyl, n-butenyl, i-butenyl, 3-methylbut-2-enyl, n-pentenyl, heptenyl, octenyl and decenyl.

"Alkoxy" means an alkyl-O- group in which the alkyl group is as described herein. Exemplary alkoxy groups include methoxy, ethoxy, n-propoxy, i-propoxy, n-butoxy and heptoxy.

"Alkyloxycarbonyl" means an alkyl-O-C(=O)- group in which the alkyl group is as described herein. Exemplary alkyloxycarbonyl groups include methoxy- and ethoxycarbonyl.

"Alkyl" means, unless otherwise specified, an aliphatic hydrocarbon group which may be straight or branched having about 1 to about 15 carbon atoms in the chain optionally substituted by alkoxy or by one or more halogen atoms. Particular alkyl groups have from 1 to about 6 carbon atoms. "Lower alkyl" as a group or part of a lower alkoxy, lower alkylthio, lower alkylsulfinyl or lower alkylsulfonyl group means unless otherwise specified, an aliphatic hydrocarbon group which may be straight or branched having about 1 to about 4 carbon atoms

in the chain. Exemplary alkyl groups include methyl, ethyl, n-propyl, i-propyl, n-butyl, s-butyl, t-butyl, n-pentyl, 3-pentyl, heptyl, octyl, nonyl, decyl and dodecyl.

"Alkylcarbonylamino" means an alkyl-C(=O)-NH- group in which the alkyl group is as described herein. Exemplary alkylcarbonylamino groups include acetamido and propionamido.

"Alkylene" means an aliphatic bivalent radical derived from a straight or branched alkyl group, in which the alkyl group is as described herein. Exemplary alkylene radicals include methylene, ethylene and trimethylene.

"Alkylenedioxy" means an -O-alkyl-O- group in which the alkyl group is as defined above. Exemplary alkylenedioxy groups include methylenedioxy and ethylenedioxy.

"Alkylsulfinyl" means an alkyl-SO- group in which the alkyl group is as previously described. Preferred alkylsulfinyl groups are those in which the alkyl group is C_{1-4}alkyl.

"Alkylsulfonyl" means an alkyl-SO_2- group in which the alkyl group is as previously described. Preferred alkylsulfonyl groups are those in which the alkyl group is C_{1-4}alkyl.

"Alkylsulfonylamino" means an alkyl-SO_2-NH- group in which the alkyl group is as described herein. Exemplary alkylsulfonylamino groups include methanesulfonamido and ethanesulfonamido.

"Alkylthio" means an alkyl-S- group in which the alkyl group is as previously described. Exemplary alkylthio groups include methylthio, ethylthio, isopropylthio and heptylthio.

"Alkynyl" means an aliphatic hydrocarbon group containing a carbon-carbon triple bond and which may be straight or branched having about 2 to about 15 carbon atoms in the chain. Preferred alkynyl groups have 2 to about 12 carbon atoms in the chain; and more preferably about 2 to about 4 carbon atoms in the chain. Exemplary alkynyl groups include ethynyl, propynyl, n-butynyl, 2-butynyl, 3-methylbut-2-ynyl, and n-pentynyl.

"Aroyl" means an aryl-CO- group in which the aryl group is as described herein. Exemplary aroyl groups include benzoyl and 1- and 2-naphthoyl.

"Aroylamino" is an aroyl-NH- group wherein aroyl is as previously defined.

"Aryl" as a group or part of a group denotes: (i) an optionally substituted monocyclic or multicyclic aromatic carbocyclic moiety of about 6 to about 14 carbon atoms, such as phenyl or naphthyl; or (ii) an optionally substituted partially saturated multicyclic aromatic carbocyclic moiety in which an aryl and a cycloalkyl or cycloalkenyl group are fused together to form a cyclic structure, such as a tetrahydronaphthyl, indenyl or indanyl ring. Aryl groups may be substituted with one or more aryl group substituents which may be the same or different, where "aryl group substituent" includes, for example, acyl, acylamino, alkoxy, alkyloxycarbonyl, alkylenedioxy, alkylsulfinyl, alkylsulfonyl, alkylthio, aroyl, aroylamino, aryl, arylalkenyl, arylalkynyl, arylalkyloxy, arylalkyloxycarbonyl, arylalkylthio, aryloxy, aryloxyalkyl, aryloxycarbonyl, arylsulfinyl, arylsulfonyl, arylthio, carboxy, cyano, halo, heteroaroyl, heteroaryl, heteroarylalkenyl, heteroarylalkynyl, heteroarylalkyloxy, heteroaroylamino, heteroaryloxy, heteroaryloxyalkyl, hydroxy, nitro, trifluoromethyl, $-NY^1Y^2$, $-CONY^1Y^2$, $-SO_2NY^1Y^2$, $-Z^2-C_{2-6}$alkylene-NY^1Y^2 {where Z^2 is O, NR^8 or $S(O)_m$}, $-NY^1$-(C=O)alkyl, $-NY^1-SO_2$alkyl or alkyl optionally substituted with alkoxy, aroyl, aryl, aryloxy, heteroaryl, hydroxy, or $-NY^1Y^2$.

"Arylalkenyl" means an aryl-alkenyl- group in which the aryl and alkenyl are as previously described. Preferred arylalkenyls contain a lower alkenyl moiety. Exemplary arylalkenyl groups include styryl and phenylallyl.

"Arylalkyl" means an aryl-alkyl- group in which the aryl and alkyl moieties are as previously described. Preferred arylalkyl groups contain a C_{1-4}alkyl moiety. Exemplary arylalkyl groups include benzyl, 2-phenethyl and naphthlenemethyl.

"Arylalkyloxy" means an arylalkyl-O- group in which the arylalkyl groups is as previously described. Exemplary arylalkyloxy groups include benzyloxy and 1- or 2-naphthalenemethoxy.

"Arylalkyloxycarbonyl" means an arylalkyl-O-CO- group in which the arylalkyl groups is as previously described. An exemplary arylalkyloxycarbonyl group is benzyloxycarbonyl.

"Arylalkylthio" means an arylalkyl-S- group in which the arylalkyl group is as previously described. An exemplary arylalkylthio group is benzylthio.

"Arylalkynyl" means an aryl-alkynyl- group in which the aryl and alkynyl are as previously described. Exemplary arylalkynyl groups include phenylethynyl and 3-phenylbut-2-ynyl.

"Aryloxy" means an aryl-O- group in which the aryl group is as previously described. Exemplary aryloxy groups include optionally substituted phenoxy and naphthoxy.

"Aryloxyalkyl" means an aryl-O-alkyl- group in which the aryl and alkyl groups are as previously described. Exemplary aryloxyalkyl groups include phenoxymethyl and 1- or 2-naphthyloxymethyl.

"Aryloxycarbonyl" means an aryl-O-C(=O)- group in which the aryl group is as previously described. Exemplary aryloxycarbonyl groups include phenoxycarbonyl and naphthoxycarbonyl.

"Arylsulfinyl" means an aryl-SO- group in which the aryl group is as previously described.

"Arylsulfonyl" means an aryl-SO_2- group in which the aryl group is as previously described.

"Arylthio" means an aryl-S- group in which the aryl group is as previously described. Exemplary arylthio groups include phenylthio and naphthylthio.

"Azaheteroaryl" means an aromatic carbocyclic moiety of about 5 to about 10 ring members in which one of the ring members is nitrogen and the other ring members are chosen from carbon, oxygen, sulfur, or nitrogen. Examples of azaheteroaryl groups include benzimidazolyl, imidazolyl, isoquinolinyl, isoxazolyl, pyrazolopyrimidinyl, pyridyl, pyrimidinyl, quinolinyl, quinazolinyl and thiazolyl.

"Cyclic amine" means a 3 to 8 membered monocyclic cycloalkyl ring system where one of the ring carbon atoms is replaced by nitrogen and which (i) may optionally contain an additional heteroatom selected from O, S or NY^3 (where Y^3 is hydrogen, alkyl, arylalkyl, and aryl) and (ii) may be fused to additional aryl or heteroaryl ring to form a bicyclic ring system. Exemplary cyclic amines include pyrrolidine, piperidine, morpholine, piperazine, indoline and pyrindoline.

"Cycloalkenyl" means a cycloalkyl group containing at least one carbon-carbon double bond. Exemplary monocyclic cycloalkenyl rings include cyclopentenyl, cyclohexenyl or cycloheptenyl.

"Cycloalkyl" means a saturated monocyclic or bicyclic ring system of about 3 to about 10 carbon atoms optionally substituted by oxo, alkyl, aryl or -C(=O)- NY^1Y^2. Exemplary monocyclic cycloalkyl rings include C_{3-8}cycloalkyl rings such as cyclopropyl, cyclopentyl, cyclohexyl and cycloheptyl.

"Cycloalkylalkyl" means a cycloalkyl-alkyl- group in which the cycloalkyl and alkyl moieties are as previously described. Exemplary monocyclic cycloalkylalkyl groups include cyclopropylmethyl, cyclopentylmethyl, cyclohexylmethyl and cycloheptylmethyl.

"Halo" or "halogen" means fluoro, chloro, bromo, or iodo. Preferred are fluoro or chloro.

"Heteroaroyl" means a heteroaryl-C(=O)- group in which the heteroaryl group is as described herein. Exemplary groups include pyridylcarbonyl.

"Heteroaroylamino" means a heteroaroyl-NH- group in which the heteroaryl moiety are as previously described.

"Heteroaryl" as a group or part of a group denotes: (i) an optionally substituted aromatic monocyclic or multicyclic organic moiety of about 5 to about 10 ring members in which one or more of the ring members is/are element(s) other than carbon, for example nitrogen, oxygen or sulfur (examples of such groups include benzimidazolyl, benzthiazolyl, benzthiophenyl, furyl, imidazolyl, indolyl, indolizinyl, isoxazolyl, isoquinolinyl, isothiazolyl, oxadiazolyl, pyrazinyl, pyridazinyl, pyrazolyl, pyridyl, pyrimidinyl, pyrrolyl, quinazolinyl, quinolinyl, 1,3,4-thiadiazolyl, thiazolyl, thienyl and triazolyl groups, optionally substituted by one or more aryl group substituents as defined above); (ii) an optionally substituted partially saturated multicyclic heterocarbocyclic moiety in which a heteroaryl and a cycloalkyl or cycloalkenyl group are fused together to form a cyclic structure (examples of such groups include pyrindanyl groups). Optional substituents include one or more "aryl group substituents" as defined above.

"Heteroarylalkenyl" means a heteroaryl-alkenyl- group in which the heteroaryl and alkenyl moieties are as previously described. Preferred heteroarylalkenyl groups contain a lower alkenyl moiety. Exemplary heteroarylalkenyl groups include pyridylethenyl and pyridylallyl.

"Heteroarylalkyl" means a heteroaryl-alkyl- group in which the heteroaryl and alkyl moieties are as previously described. Preferred heteroarylalkyl groups contain a C_{1-4}alkyl moiety. Exemplary heteroarylalkyl groups include pyridylmethyl.

"Heteroarylalkyloxy" means an heteroarylalkyl-O- group in which the heteroarylalkyl group is as previously described. Exemplary heteroaryloxy groups include optionally substituted pyridylmethoxy.

"Heteroarylalkynyl" means a heteroaryl-alkynyl- group in which the heteroaryl and alkynyl moieties are as previously described. Exemplary heteroarylalkenyl groups include pyridylethynyl and 3-pyridylbut-2-ynyl.

"Heteroaryloxy" means an heteroaryl-O- group in which the heteroaryl group is as previously described. Exemplary heteroaryloxy groups include optionally substituted pyridyloxy.

"Heteroaryloxyalkyl" means an heteroaryl-O-alkyl- group in which the heteroaryl and alkyl groups are as previously described. Exemplary heteroaryloxyalkyl groups include pyridyoxymethyl and 2-, 3- or 4-quinolinyloxymethyl.

"Heterocycloalkenyl" means a cycloalkenyl group of about 3 to 7 ring members which contains one or more heteroatoms selected from O, S or NY^4 (where Y^4 is hydrogen, alkyl, aryl, arylalkyl, and alkyloxycarbonyl). Exemplary heterocycloalkenyl groups include 1,2,3,6-tetrahydro-pyridine.

"Heterocycloalkyl" means: (i) a cycloalkyl group of about 3 to 7 ring members which contains one or more heteroatoms selected from O, S or NY^4 (where Y^4 is hydrogen, alkyl, aryl, arylalkyl, and alkyloxycarbonyl) and which may optionally be substituted by oxo (examples of such groups include piperidinyl, pyrrolidinyl, morpholinyl, tetrahydropyranyl and tetrahydrothiophenyl; (ii) an optionally substituted partially saturated multicyclic heterocarbocyclic moiety in which one or more aryl (or heteroaryl) rings and a cycloalkyl group of about 3 to 7 ring members, which contains one or more heteroatoms selected from O, S or NY^4 and which may optionally be substituted by oxo, are fused together to form a cyclic structure (examples of such groups include chromanyl, dihydrobenzofuranyl, indolinyl and pyrindolinyl groups).

"Heterocycloalkylalkyl" means a heterocycloalkyl-alkyl- group in which the heterocycloalkyl and alkyl moieties are as previously described.

"Heterocycloalkylalkyloxy" means a heterocycloalkyl-alkyl-O- group in which the heterocycloalkyl and alkyl moieties are as previously described.

"Heterocycloalkyloxy" means a heterocycloalkyl-O- group in which the heterocycloalkyl is as previously described.

"Prodrug" means a compound which is suitable for administration to a patient without undue toxicity, irritation, allergic response, and the like, and is convertible in vivo by metabolic means (e.g. by hydrolysis) to a compound of formula (I), including N-oxides thereof. A thorough discussion is provided in T. Higuchi and V. Stella, Pro-drugs as Novel Delivery Systems, Vol. 14 of the A. C. S. Symposium Series, and in Edward B. Roche, ed., Bioreversible Carriers in Drug Design, American Pharmaceutical Association and Pergamon Press, 1987, both of which are incorporated herein by reference. For example an ester of a compound of formula (I) containing a hydroxy group may be convertible by hydrolysis in vivo to the parent molecule. Alternatively an ester of a compound of formula (I) containing a carboxy group may be convertible by hydrolysis in vivo to the parent molecule.

Suitable esters of compounds of formula (I) containing a hydroxy group, are for example acetates, citrates, lactates, tartrates, malonates, oxalates, salicylates, propionates, succinates, fumarates, maleates, methylene-bis-β-hydroxynaphthoates, gentisates, isethionates, di-p-toluoyltartrates, methanesulfonates, ethanesulfonates, benzenesulfonates, p-toluenesulfonates, cyclohexylsulfamates and quinates.

An especially useful class of esters of compounds of formula (I) containing a hydroxy group, may be formed from acid moieties selected from those described by Bundgaard et. al., J. Med. Chem., 1989, 32, page 2503-2507, and include substituted (aminomethyl)-benzoates, for example dialkylamino-methylbenzoates in which the two alkyl groups may be joined together and/or interrupted by an oxygen atom or by an optionally substituted nitrogen atom, e.g. an alkylated nitrogen atom, more especially (morpholino-methyl)benzoates, e.g. 3- or 4-(morpholinomethyl)-benzoates, and (4-alkylpiperazin-1-yl)benzoates, e.g. 3- or 4-(4-alkylpiperazin-1-yl)benzoates.

Suitable esters of compounds of formula (I) containing a carboxy group, are for example those described by F.J.Leinweber, Drug Metab. Res., 1987, 18, page 379.

The compounds of the present invention are basic, and such compounds are useful in the form of the free base or in the form of a pharmaceutically acceptable acid addition salt thereof.

Acid addition salts are a more convenient form for use; and in practice, use of the salt form inherently amounts to use of the free base form. The acids which can be used to prepare the acid addition salts include preferably those which produce, when combined with the free base, pharmaceutically acceptable salts, that is, salts whose anions are non-toxic to the patient in pharmaceutical doses of the salts, so that the beneficial inhibitory effects inherent in the free base are not vitiated by side effects ascribable to the anions. Although pharmaceutically acceptable salts of said basic compounds are preferred, all acid addition salts are useful as sources of the free base form even if the particular salt, per se, is desired only as an intermediate product as, for example, when the salt is formed only for purposes of purification, and identification, or when it is used as intermediate in preparing a pharmaceutically acceptable salt by ion exchange procedures. Pharmaceutically acceptable salts within the scope of the invention include those derived from mineral acids and organic acids, and include hydrohalides, e.g. hydrochlorides and hydrobromides, sulfates, phosphates, nitrates, sulfamates, acetates, citrates, lactates, tartrates, malonates, oxalates, salicylates, propionates, succinates, fumarates, maleates, methylene-bis-b-hydroxynaphthoates, gentisates, isethionates, di-p-toluoyltartrates, methane-sulfonates, ethanesulfonates, benzenesulfonates, p-toluenesulfonates, cyclohexylsulfamates and quinates.

As well as being useful in themselves as active compounds, salts of compounds of the invention are useful for the purposes of purification of the compounds, for example by exploitation of the solubility differences between the salts and the parent compounds, side products and/or starting materials by techniques well known to those skilled in the art.

With reference to formula (I) above, the following are particular and preferred groupings:

R^1 may particularly represent hydrogen.

R^2 may particularly represent hydrogen.

R^3 may particularly represent aryl, such as optionally substituted phenyl or optionally substituted naphthyl, especially substituted phenyl. Exemplary optional substituents include one or more halo atoms or alkyl substituted by aryl, alkyl substituted by aryloxy, alkyl substituted by aroyl, alkyl substituted by heteroaryl, arylalkynyl, heteroarylalkynyl, aryl, heteroaryl, arylalkenyl or arylalkyloxy, in which the aryl or heteroaryl groups may be further substituted by one or more aryl group substituents.

R^3 may also particularly represent heteroaryl, such as optionally substituted pyridyl, optionally substituted quinolinyl, optionally substituted thienyl, optionally substituted furanyl or optionally substituted indolyl, especially substituted thienyl, substituted pyridyl or indolyl. Exemplary optional substituents include alkyl substituted by aryl, alkyl substituted by aryloxy, alkyl substituted by aroyl, alkyl substituted heteroaryl, arylalkynyl, heteroarylalkynyl, heteroaryl, arylalkenyl or arylalkyloxy, in which the aryl or heteroaryl groups are further substituted by one or more aryl group substituents.

R^4 may particularly represent hydrogen.

R^4 may also particularly represent cyano, especially when attached to the tertiary ring carbon atom.

R^5 may particularly represent hydrogen.

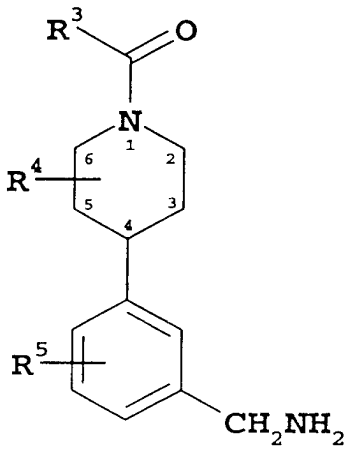
R^5 may also particularly represent lower alkyl (e.g. methyl) or halo (e.g. fluoro).

 may particularly represent a single bond.

n may particularly represent 2.

It is to be understood that this invention covers all appropriate combinations of the particular and preferred groupings referred to herein.

A particular group of compounds of the invention are compounds of formula (Ia):-



(Ia)

in which R^3, R^4 and R^5 are as hereinbefore defined and the corresponding N-oxides, and their prodrugs; and pharmaceutically acceptable salts and solvates (e.g. hydrates) of such compounds and their N-oxides and prodrugs.

Compounds of formula (Ia) in which R^3 represents aryl, such as optionally substituted phenyl or optionally substituted naphthyl, especially substituted phenyl, are preferred. Preferred optional substituents include one or more halo atoms or alkyl substituted by aryl or alkyl substituted heteroaryl, in which the aryl or heteroaryl groups may be further substituted by one or more aryl group substituents. R^3 especially represents dichlorophenyl [e.g. 3,4-dichlorophenyl], phenylC_{1-3}alkylphenyl [e.g. phenethyl], hydroxyphenylC_{1-3}alkylphenyl [e.g. 4-hydroxyphenylethylphenyl] and aminopyridylC_{1-3}alkylphenyl [e.g. (4-amino-pyrid-3-yl)ethylphenyl].

Compounds of formula (Ia) in which R^3 represents heteroaryl, such as optionally substituted pyridyl, optionally substituted quinolinyl, optionally substituted thienyl, optionally substituted furanyl or optionally substituted indolyl, especially substituted thienyl, substituted pyridyl or indolyl, are preferred. Preferred optional substituents include alkyl substituted by aryl and alkyl substituted heteroaryl in which the aryl or heteroaryl groups are further substituted by one or more aryl group substituents. R^3 especially represents phenylC_{1-3}alkylpyridyl [e.g. 5-phenylethyl-pyrid-3-yl], phenylC_{1-3}alkylthienyl [e.g. 5-phenylethyl-thien-2-yl] and indolyl [e.g. indol-6-yl].

Compounds of formula (Ia) in which R^4 represents hydrogen are preferred.

Compounds of formula (Ia) in which R^4 represents cyano are also preferred. R^4 is preferably attached at the 4 position of the piperidine ring.

Compounds of formula (Ia) in which R^5 represents hydrogen are preferred.

Compounds of formula (Ia) in which R^5 represents lower alkyl (e.g. methyl) or halo (e.g. fluoro), are also preferred. R^5 is preferably attached to the phenyl ring in the position para to the $-CH_2NH_2$ group.

A preferred group of compounds of the invention are compounds of formula (Ia) in which:- R^3 is substituted phenyl [especially 3,4-dichlorophenyl, phenethyl, 4-hydroxyphenylethylphenyl and (4-amino-pyrid-3-yl)ethylphenyl] or optionally substituted heteroaryl [especially 5-phenylethyl-pyrid-3-yl, 5-phenylethyl-thien-2-yl or indol-6-yl]; R^4 is hydrogen, or cyano attached at the 4 position of the piperidine ring; R^5 is hydrogen, or lower alkyl (e.g. methyl) or halo (e.g. fluoro) attached to the phenyl ring in the position para to the $-CH_2NH_2$ group; and the corresponding N-oxides, and their prodrugs; and pharmaceutically acceptable salts and solvates (e.g. hydrates) of such compounds and their N-oxides and prodrugs.

Particular compounds of the invention are selected from the compounds formed by joining the carbon atom (C*) of one of the fragments (A1 to A10) shown in Table 1 to the carbon atom (C*) of one of the fragments (B1 to B12) shown in Table 2, and joining the nitrogen atom (N*) of one of the fragments (B1 to B12) shown in Table 2 to the carbon atom (C*) of one of the acidic fragments (C1 to C103) depicted in Table 3.

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TABLE 1

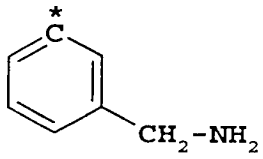
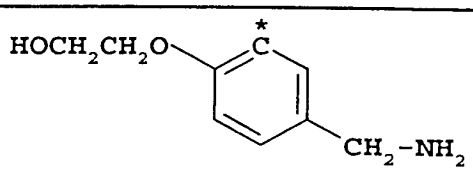
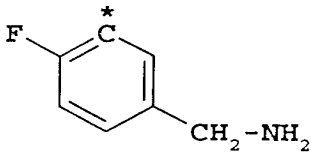
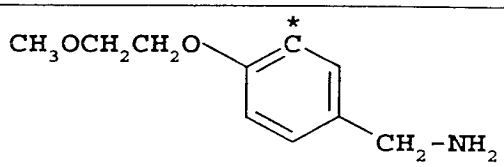
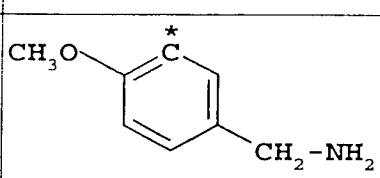
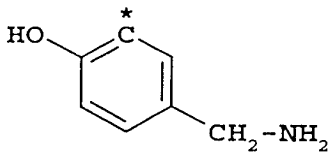
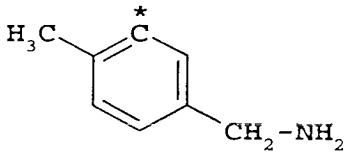
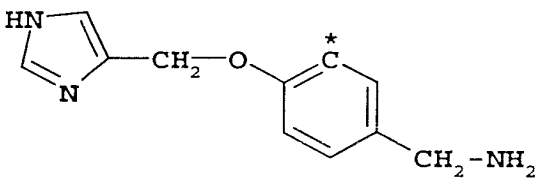
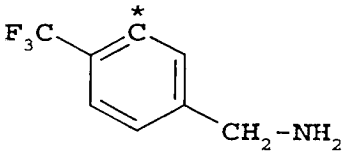
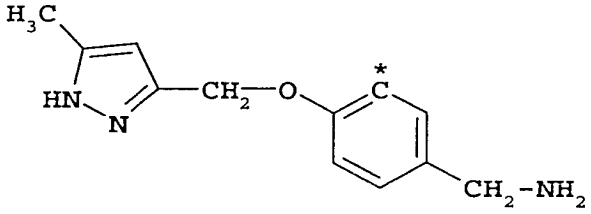
A1		A2	
A3		A4	
A5		A6	
A7		A8	
A9		A10	

TABLE 2

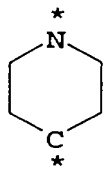
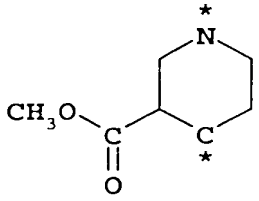
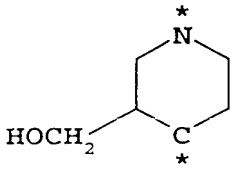
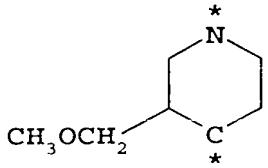
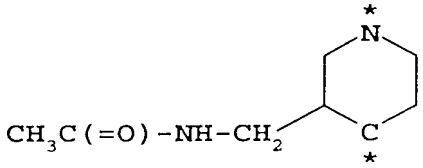
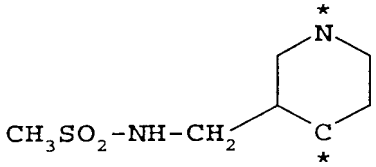
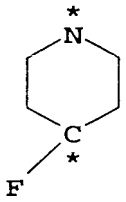
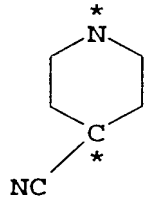
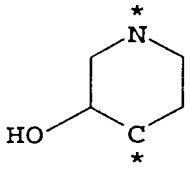
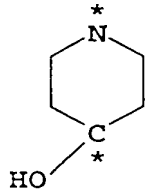
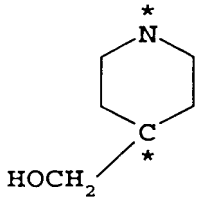
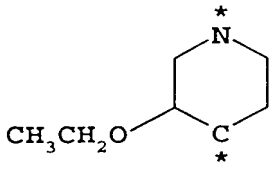
B1		B2	
B3		B4	
B5		B6	
B7		B8	
B9		B10	
B11		B12	

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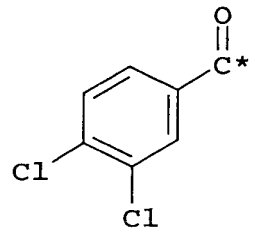
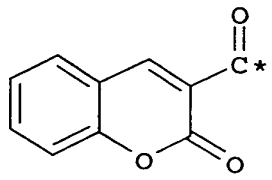
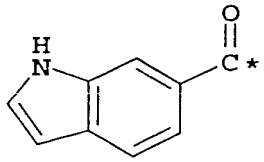
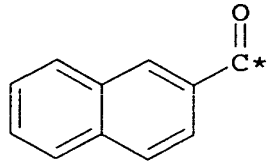
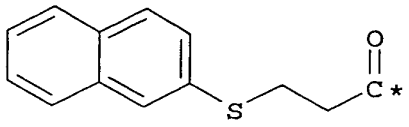
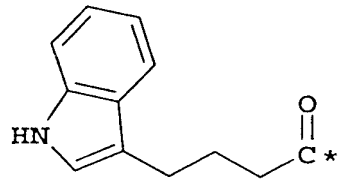
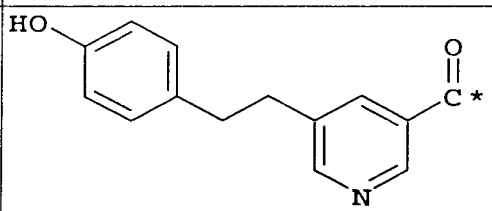
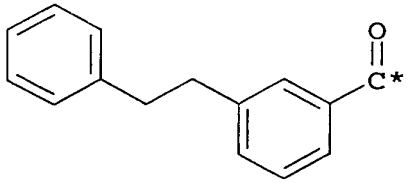
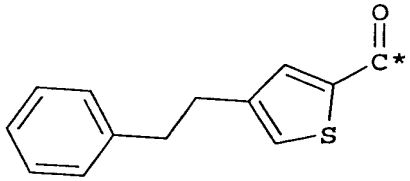
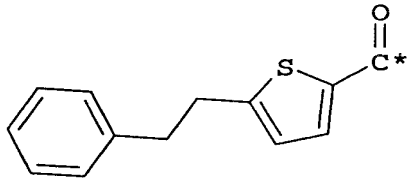
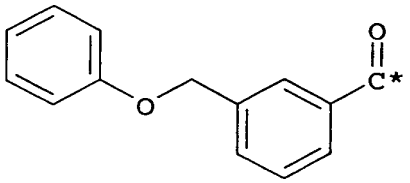
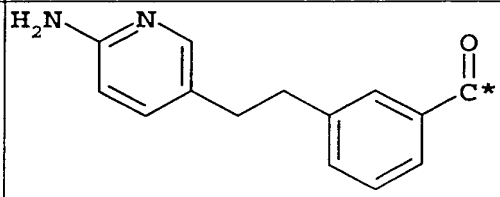
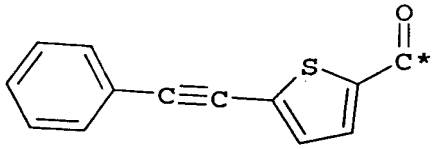
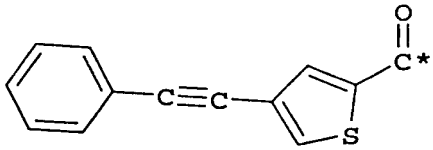
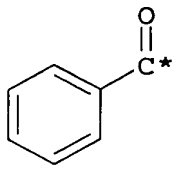
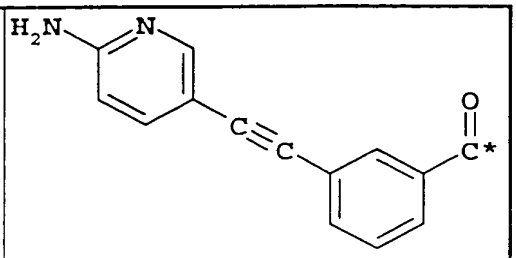
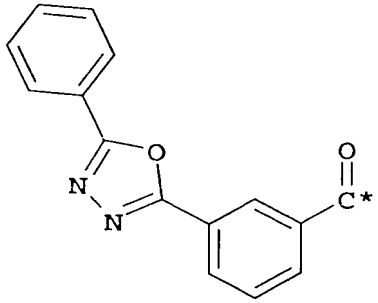
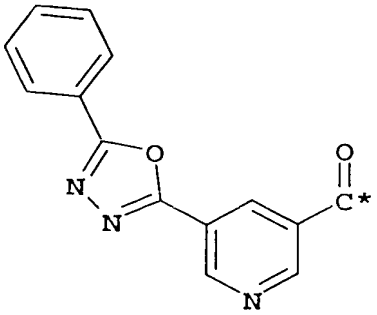
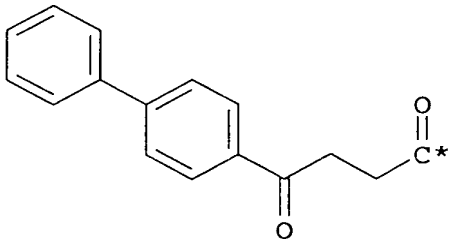
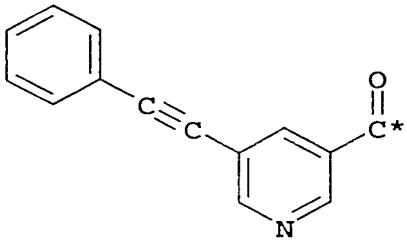
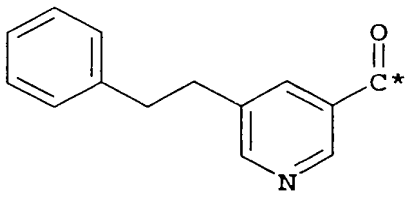
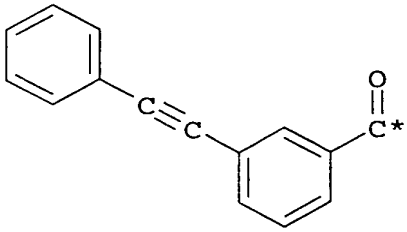
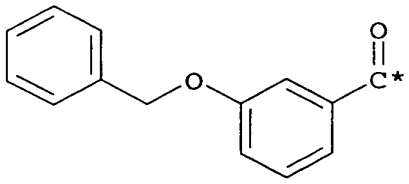
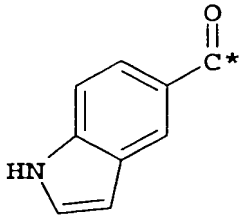
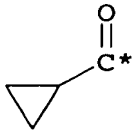
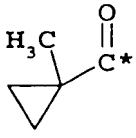
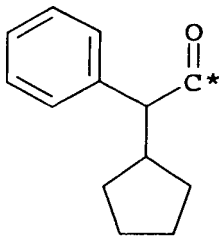
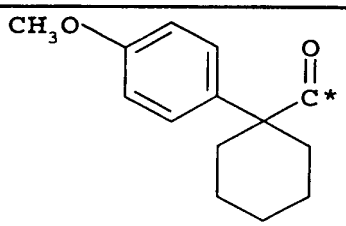
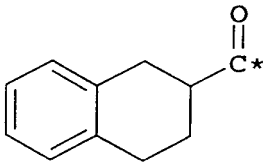
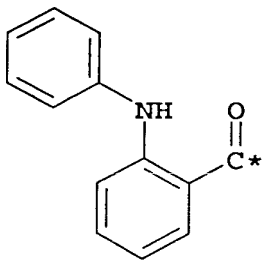
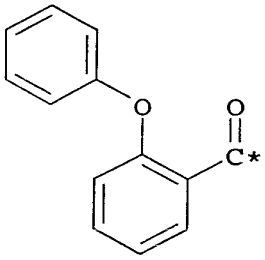
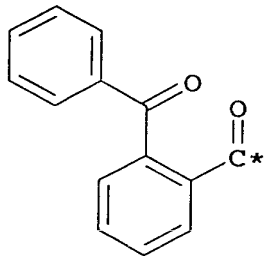
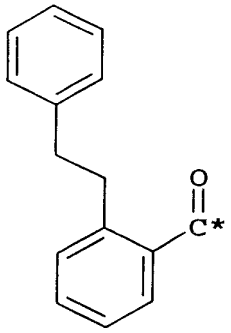
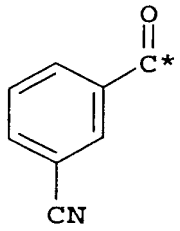
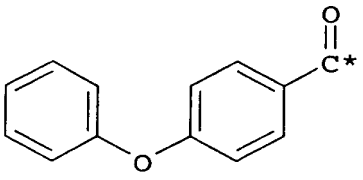
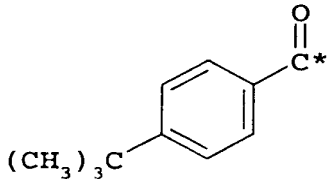
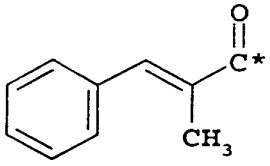
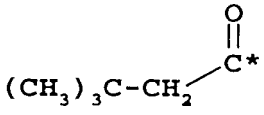
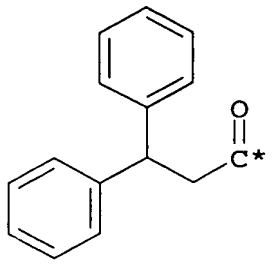
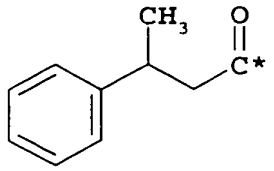
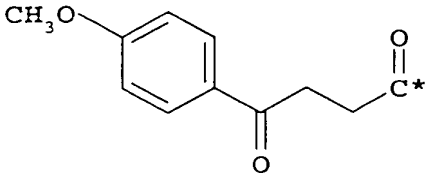
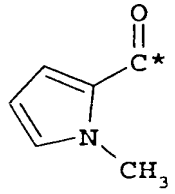
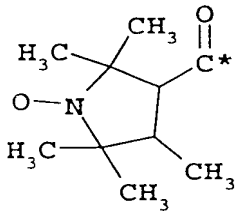
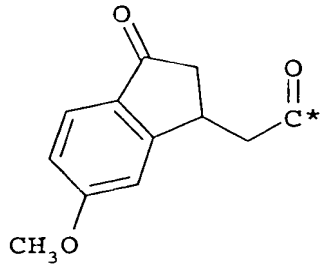
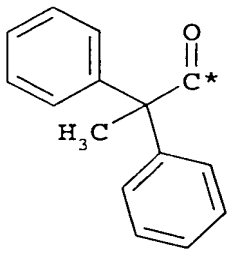
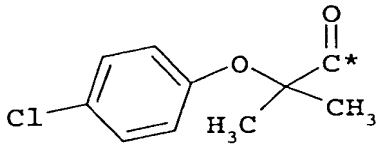
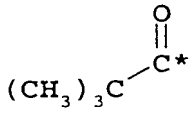
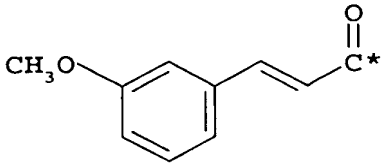
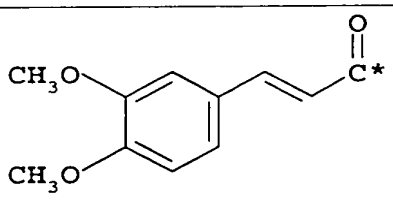
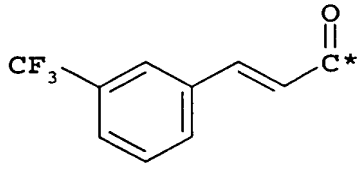
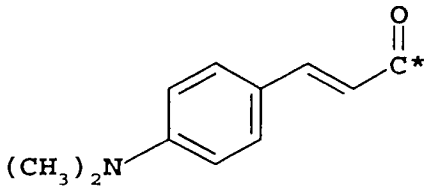
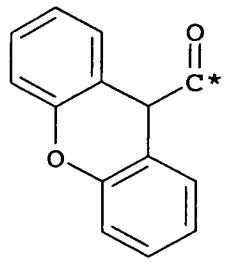
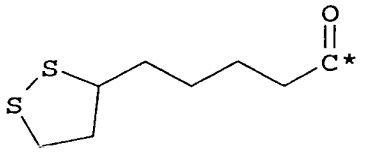
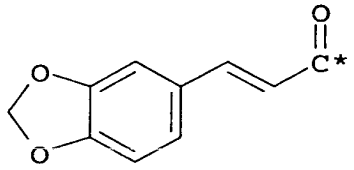
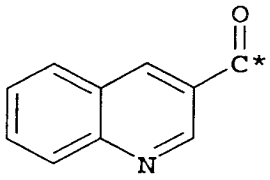
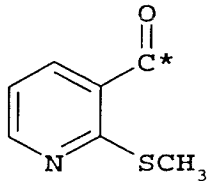
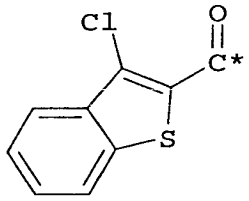
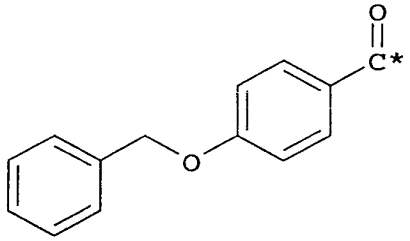
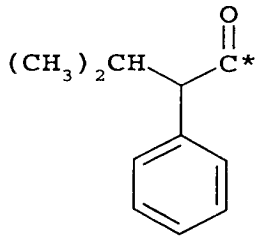
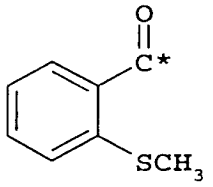
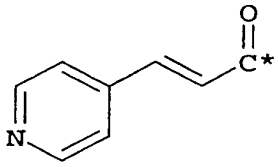
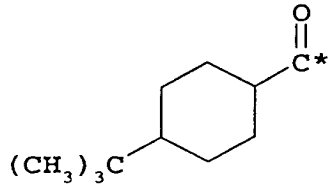
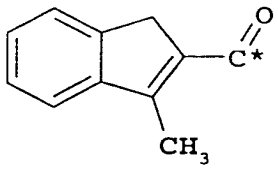
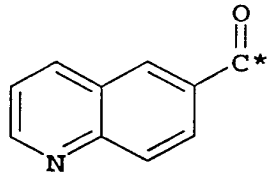
TABLE 3

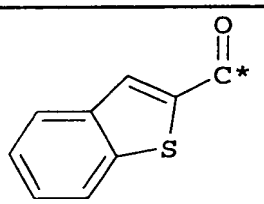
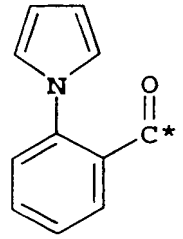
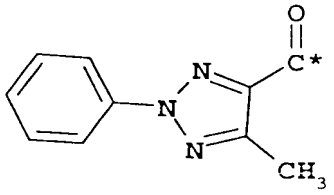
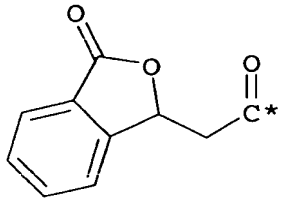
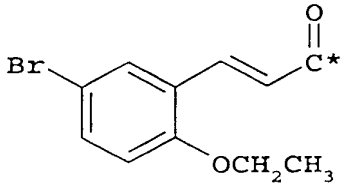
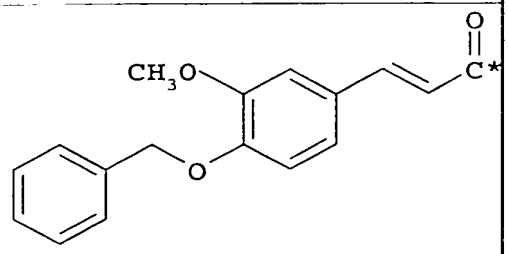
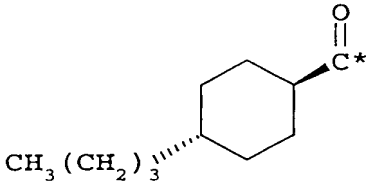
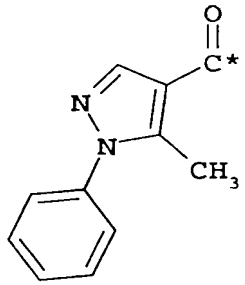
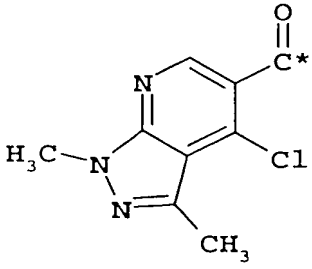
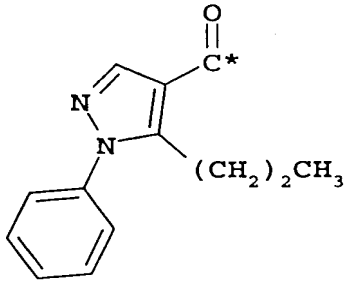
C1		C2	
C3		C4	
C5		C6	
C7		C8	
C9		C10	
C11		C12	
C13		C14	

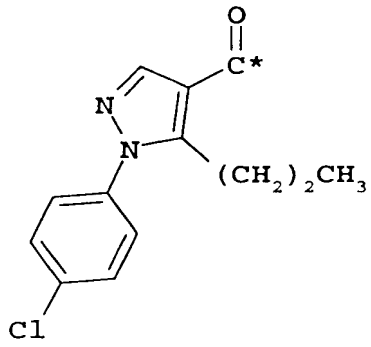
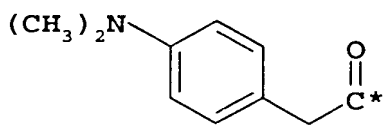
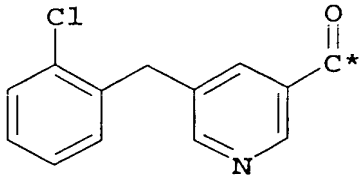
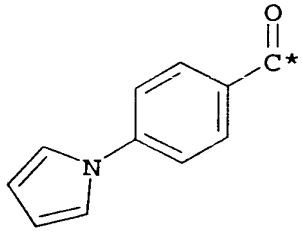
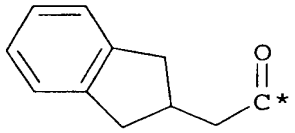
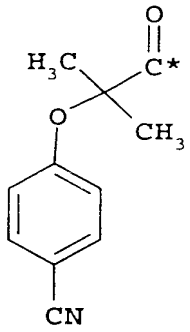
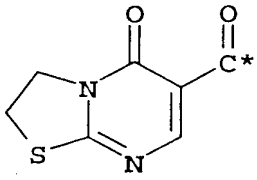
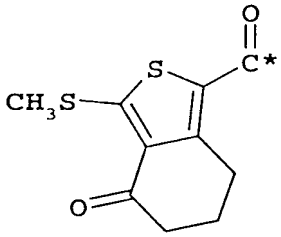
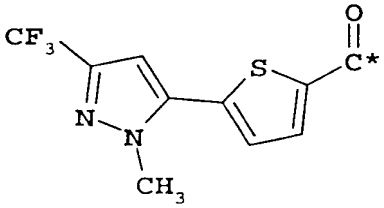
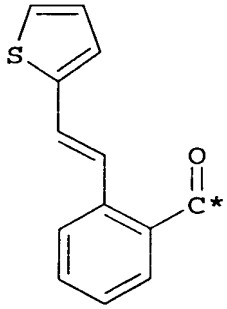
C15		C16	
C17		C18	
C19		C20	
C21		C22	
C23		C24	
C25		C26	

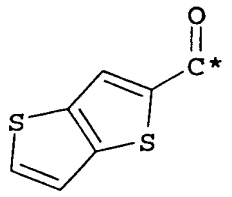
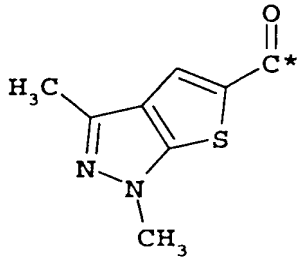
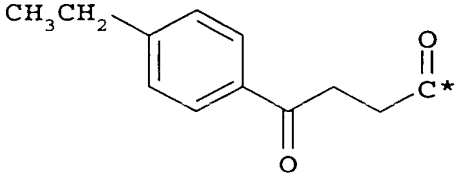
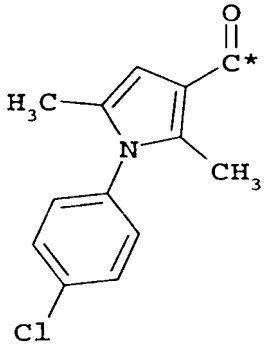
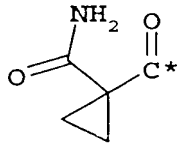
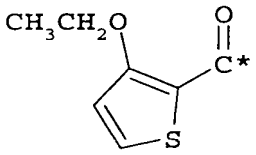
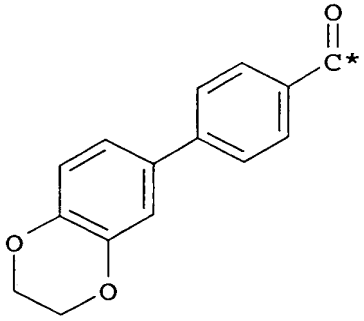
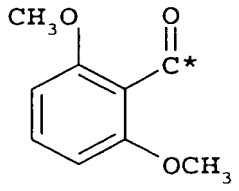
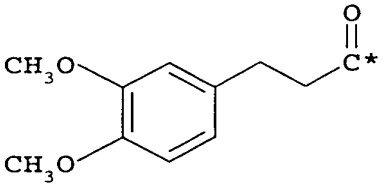
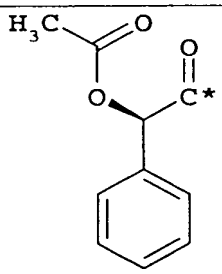
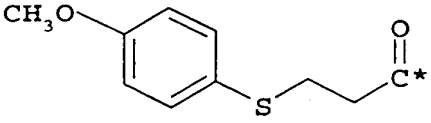
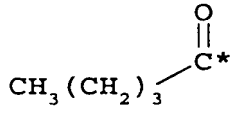
C27		C28	
C29		C30	
C31		C32	
C33		C34	
C35		C36	
C37		C38	

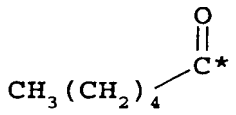
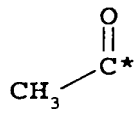
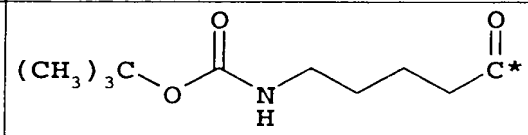
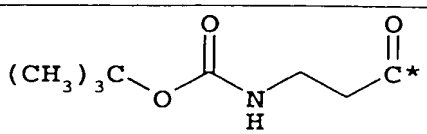
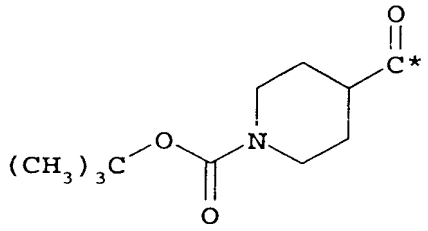
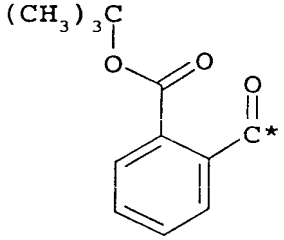
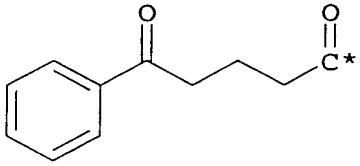
C39		C40	
C41		C42	
C43		C44	
C45		C46	
C47		C48	
C49		C50	

C51		C52	
C53		C54	
C55		C56	
C57		C58	
C59		C60	
C61		C62	
C63		C64	

C65		C66	
C67		C68	
C69		C70	
C71		C72	
C73		C74	

C75		C76	
C77		C78	
C79		C80	
C81		C82	
C83		C84	

C85		C86	
C87		C88	
C89		C90	
C91		C92	
C93		C94	
C95		C96	

C97		C98	
C99		C100	
C101		C102	
C103			

Particularly preferred examples of fragments "A", "B", and "C" are illustrated below:

A1-B1-C1;	A1-B1-C2;	A1-B1-C3;	A1-B1-C4;	A1-B1-C5;	A1-B1-C6;
A1-B1-C7;	A1-B1-C8;	A1-B1-C9;	A1-B1-C10;	A1-B1-C11;	A1-B1-C12;
A1-B1-C13;	A1-B1-C14;	A1-B1-C15;	A1-B1-C16;	A1-B1-C17;	A1-B1-C18;
A1-B1-C19;	A1-B1-C20;	A1-B1-C21;	A1-B1-C22;	A1-B1-C23;	A1-B1-C24;
A1-B1-C25;	A1-B1-C26;	A1-B1-C27;	A1-B1-C28;	A1-B1-C29;	A1-B1-C30;
A1-B1-C31;	A1-B1-C32;	A1-B1-C33;	A1-B1-C34;	A1-B1-C35;	A1-B1-C36;
A1-B1-C37;	A1-B1-C38;	A1-B1-C39;	A1-B1-C40;	A1-B1-C41;	A1-B1-C42;
A1-B1-C43;	A1-B1-C44;	A1-B1-C45;	A1-B1-C46;	A1-B1-C47;	A1-B1-C48;
A1-B1-C49;	A1-B1-C50;	A1-B1-C51;	A1-B1-C52;	A1-B1-C53;	A1-B1-C54;
A1-B1-C55;	A1-B1-C56;	A1-B1-C57;	A1-B1-C58;	A1-B1-C59;	A1-B1-C60;
A1-B1-C61;	A1-B1-C62;	A1-B1-C63;	A1-B1-C64;	A1-B1-C65;	A1-B1-C66;
A1-B1-C67;	A1-B1-C68;	A1-B1-C69;	A1-B1-C70;	A1-B1-C71;	A1-B1-C72;
A1-B1-C73;	A1-B1-C74;	A1-B1-C75;	A1-B1-C76;	A1-B1-C77;	A1-B1-C78;
A1-B1-C79;	A1-B1-C80;	A1-B1-C81;	A1-B1-C82;	A1-B1-C83;	A1-B1-C84;
A1-B1-C85;	A1-B1-C86;	A1-B1-C87;	A1-B1-C88;	A1-B1-C89;	A1-B1-C90;
A1-B1-C91;	A1-B1-C92;	A1-B1-C93;	A1-B1-C94;	A1-B1-C95;	A1-B1-C96;

A1-B1-C97; A1-B1-C98; A1-B1-C99; A1-B1-C100; A1-B1-C101; A1-B1-C102; A1-B1-C103; A1-B2-C1; A1-B2-C2; A1-B2-C3; A1-B2-C4; A1-B2-C5; A1-B2-C6; A1-B2-C7; A1-B2-C8; A1-B2-C9; A1-B2-C10; A1-B2-C11; A1-B2-C12; A1-B2-C13; A1-B2-C14; A1-B2-C15; A1-B2-C16; A1-B2-C17; A1-B2-C18; A1-B2-C19; A1-B2-C20; A1-B2-C21; A1-B2-C22; A1-B2-C23; A1-B2-C24; A1-B2-C25; A1-B2-C26; A1-B2-C27; A1-B2-C28; A1-B2-C29; A1-B2-C30; A1-B2-C31; A1-B2-C32; A1-B2-C33; A1-B2-C34; A1-B2-C35; A1-B2-C36; A1-B2-C37; A1-B2-C38; A1-B2-C39; A1-B2-C40; A1-B2-C41; A1-B2-C42; A1-B2-C43; A1-B2-C44; A1-B2-C45; A1-B2-C46; A1-B2-C47; A1-B2-C48; A1-B2-C49; A1-B2-C50; A1-B2-C51; A1-B2-C52; A1-B2-C53; A1-B2-C54; A1-B2-C55; A1-B2-C56; A1-B2-C57; A1-B2-C58; A1-B2-C59; A1-B2-C60; A1-B2-C61; A1-B2-C62; A1-B2-C63; A1-B2-C64; A1-B2-C65; A1-B2-C66; A1-B2-C67; A1-B2-C68; A1-B2-C69; A1-B2-C70; A1-B2-C71; A1-B2-C72; A1-B2-C73; A1-B2-C74; A1-B2-C75; A1-B2-C76; A1-B2-C77; A1-B2-C78; A1-B2-C79; A1-B2-C80; A1-B2-C81; A1-B2-C82; A1-B2-C83; A1-B2-C84; A1-B2-C85; A1-B2-C86; A1-B2-C87; A1-B2-C88; A1-B2-C89; A1-B2-C90; A1-B2-C91; A1-B2-C92; A1-B2-C93; A1-B2-C94; A1-B2-C95; A1-B2-C96; A1-B2-C97; A1-B2-C98; A1-B2-C99; A1-B2-C100; A1-B2-C101; A1-B2-C102; A1-B2-C103; A1-B3-C1; A1-B3-C2; A1-B3-C3; A1-B3-C4; A1-B3-C5; A1-B3-C6; A1-B3-C7; A1-B3-C8; A1-B3-C9; A1-B3-C10; A1-B3-C11; A1-B3-C12; A1-B3-C13; A1-B3-C14; A1-B3-C15; A1-B3-C16; A1-B3-C17; A1-B3-C18; A1-B3-C19; A1-B3-C20; A1-B3-C21; A1-B3-C22; A1-B3-C23; A1-B3-C24; A1-B3-C25; A1-B3-C26; A1-B3-C27; A1-B3-C28; A1-B3-C29; A1-B3-C30; A1-B3-C31; A1-B3-C32; A1-B3-C33; A1-B3-C34; A1-B3-C35; A1-B3-C36; A1-B3-C37; A1-B3-C38; A1-B3-C39; A1-B3-C40; A1-B3-C41; A1-B3-C42; A1-B3-C43; A1-B3-C44; A1-B3-C45; A1-B3-C46; A1-B3-C47; A1-B3-C48; A1-B3-C49; A1-B3-C50; A1-B3-C51; A1-B3-C52; A1-B3-C53; A1-B3-C54; A1-B3-C55; A1-B3-C56; A1-B3-C57; A1-B3-C58; A1-B3-C59; A1-B3-C60; A1-B3-C61; A1-B3-C62; A1-B3-C63; A1-B3-C64; A1-B3-C65; A1-B3-C66; A1-B3-C67; A1-B3-C68; A1-B3-C69; A1-B3-C70; A1-B3-C71; A1-B3-C72; A1-B3-C73; A1-B3-C74; A1-B3-C75; A1-B3-C76; A1-B3-C77; A1-B3-C78; A1-B3-C79; A1-B3-C80; A1-B3-C81; A1-B3-C82; A1-B3-C83; A1-B3-C84; A1-B3-C85; A1-B3-C86; A1-B3-C87; A1-B3-C88; A1-B3-C89; A1-B3-C90; A1-B3-C91; A1-B3-C92; A1-B3-C93; A1-B3-C94; A1-B3-C95; A1-B3-C96; A1-B3-C97; A1-B3-C98; A1-B3-C99; A1-B3-C100;

A1-B3-C101; A1-B3-C102; A1-B3-C103; A1-B4-C1; A1-B4-C2; A1-B4-C3;
A1-B4-C4; A1-B4-C5; A1-B4-C6; A1-B4-C7; A1-B4-C8; A1-B4-C9;
A1-B4-C10; A1-B4-C11; A1-B4-C12; A1-B4-C13; A1-B4-C14; A1-B4-C15;
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A9-B12-C14; A9-B12-C15; A9-B12-C16; A9-B12-C17; A9-B12-C18; A9-B12-C19; A9-B12-C20; A9-B12-C21; A9-B12-C22; A9-B12-C23; A9-B12-C24; A9-B12-C25; A9-B12-C26; A9-B12-C27; A9-B12-C28; A9-B12-C29; A9-B12-C30; A9-B12-C31; A9-B12-C32; A9-B12-C33; A9-B12-C34; A9-B12-C35; A9-B12-C36; A9-B12-C37; A9-B12-C38; A9-B12-C39; A9-B12-C40; A9-B12-C41; A9-B12-C42; A9-B12-C43; A9-B12-C44; A9-B12-C45; A9-B12-C46; A9-B12-C47; A9-B12-C48; A9-B12-C49; A9-B12-C50; A9-B12-C51; A9-B12-C52; A9-B12-C53; A9-B12-C54; A9-B12-C55; A9-B12-C56; A9-B12-C57; A9-B12-C58; A9-B12-C59; A9-B12-C60; A9-B12-C61; A9-B12-C62; A9-B12-C63; A9-B12-C64; A9-B12-C65; A9-B12-C66; A9-B12-C67; A9-B12-C68; A9-B12-C69; A9-B12-C70; A9-B12-C71; A9-B12-C72; A9-B12-C73; A9-B12-C74; A9-B12-C75; A9-B12-C76; A9-B12-C77; A9-B12-C78; A9-B12-C79; A9-B12-C80; A9-B12-C81; A9-B12-C82; A9-B12-C83; A9-B12-C84; A9-B12-C85; A9-B12-C86; A9-B12-C87; A9-B12-C88; A9-B12-C89; A9-B12-C90; A9-B12-C91; A9-B12-C92; A9-B12-C93; A9-B12-C94; A9-B12-C95; A9-B12-C96; A9-B12-C97; A9-B12-C98; A9-B12-C99; A9-B12-C100; A9-B12-C101; A9-B12-C102; A9-B12-C103; A10-B11-C1; A10-B11-C2; A10-B11-C3; A10-B11-C4; A10-B11-C5; A10-B11-C6; A10-B11-C7; A10-B11-C8; A10-B11-C9; A10-B11-C10; A10-B11-C11; A10-B11-C12; A10-B11-C13; A10-B11-C14; A10-B11-C15; A10-B11-C16; A10-B11-C17; A10-B11-C18; A10-B11-C19; A10-B11-C20; A10-B11-C21; A10-B11-C22; A10-B11-C23; A10-B11-C24; A10-B11-C25; A10-B11-C26; A10-B11-C27; A10-B11-C28; A10-B11-C29; A10-B11-C30; A10-B11-C31; A10-B11-C32; A10-B11-C33; A10-B11-C34; A10-B11-C35; A10-B11-C36; A10-B11-C37; A10-B11-C38; A10-B11-C39; A10-B11-C40; A10-B11-C41; A10-B11-C42; A10-B11-C43; A10-B11-C44; A10-B11-C45; A10-B11-C46; A10-B11-C47; A10-B11-C48; A10-B11-C49; A10-B11-C50; A10-B11-C51; A10-B11-C52; A10-B11-C53; A10-B11-C54; A10-B11-C55; A10-B11-C56; A10-B11-C57; A10-B11-C58; A10-B11-C59; A10-B11-C60; A10-B11-C61; A10-B11-C62; A10-B11-C63; A10-B11-C64; A10-B11-C65; A10-B11-C66; A10-B11-C67; A10-B11-C68; A10-B11-C69; A10-B11-C70; A10-B11-C71; A10-B11-C72; A10-B11-C73; A10-B11-C74; A10-B11-C75; A10-B11-C76; A10-B11-C77; A10-B11-C78; A10-B11-C79; A10-B11-C80; A10-B11-C81; A10-B11-C82; A10-B11-C83; A10-B11-C84; A10-B11-C85; A10-B11-C86; A10-B11-C87; A10-B11-C88; A10-B11-C89; A10-B11-C90; A10-B11-C91; A10-B11-C92; A10-B11-C93; A10-B11-C94; A10-B11-C95; A10-B11-C96; A10-B11-C97; A10-B11-C98; A10-B11-C99; A10-B11-C100; A10-B11-C101; A10-B11-C102; A10-B11-C103; A10-B12-C1; A10-B12-C2; A10-B12-C3; A10-B12-C4; A10-B12-C5; A10-B12-C6; A10-B12-C7; A10-B12-C8; A10-B12-C9; A10-B12-C10; A10-B12-C11; A10-B12-C12; A10-B12-C13; A10-B12-C14; A10-B12-C15; A10-B12-C16; A10-B12-C17;

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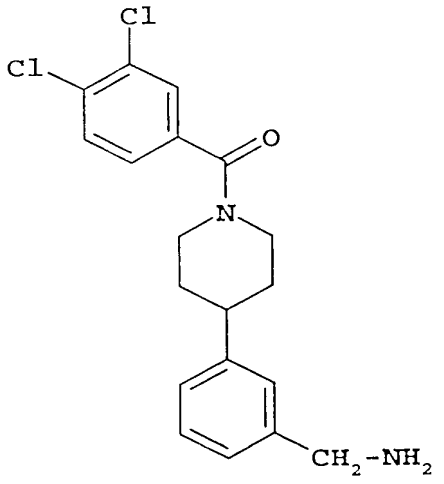
Thus, for example, in the above list the compound denoted as A1-B1-C1 is the product of the combination of group A1 in Table 1 and B1 in Table 2 and C1 in Table 3, namely



Preferred compounds of the invention are selected from:

3-[1-(5-phenylethynyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine;

3-[1-(3-phenylethyl-benzoyl)-piperidin-4-yl]-benzylamine;

3-{1-[3-(4-hydroxyphenyl)ethyl-benzoyl]-piperidin-4-yl}-benzylamine;

3-{1-[3-(6-amino-pyridin-3-yl)ethyl-benzoyl]-piperidin-4-yl}-benzylamine;

3-[1-(5-phenylethyl-thiophene-2-carbonyl)-piperidin-4-yl]-benzylamine;

4-fluoro-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine;
4-methyl-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine;
3-[1-(indole-6-carbonyl)-piperidin-4-yl]-benzylamine;
4-(3-aminomethyl-phenyl)-1-(5-phenethyl-pyridine-3-carbonyl)-piperidine-4-carbonitrile
[4-(3-aminomethylphenyl)piperidin-1-yl]-(3,4-dichlorophenyl)methanone;
and the corresponding N-oxides, and their prodrugs; and pharmaceutically acceptable salts and solvates (e.g. hydrates) of such compounds and their N-oxides and prodrugs.

The compounds of the invention exhibit useful pharmacological activity and accordingly are incorporated into pharmaceutical compositions and used in the treatment of patients suffering from certain medical disorders. The present invention thus provides, according to a further aspect, compounds of the invention and compositions containing compounds of the invention for use in therapy.

Compounds within the scope of the present invention possess tryptase inhibition activity according to tests described in the literature and described *in vitro* procedures hereinafter, and which tests results are believed to correlate to pharmacological activity in humans and other mammals. Thus, in a further embodiment, the present invention provides compounds of the invention and compositions containing compounds of the invention for use in the treatment of a patient suffering from, or subject to, conditions which can be ameliorated by the administration of an inhibitor of tryptase. For example, compounds of the present invention are useful in the treatment of inflammatory diseases, for example joint inflammation, including arthritis, rheumatoid arthritis and other arthritic conditions such as rheumatoid spondylitis, gouty arthritis, traumatic arthritis, rubella arthritis, psoriatic arthritis, osteoarthritis and other chronic inflammatory joint diseases, or diseases of joint cartilage destruction, ocular conjunctivitis, vernal conjunctivis, inflammatory bowel disease, asthma, allergic rhinitis, interstitial lung diseases, fibrosis, sceleroderma, pulmonary fibrosis, liver cirrhosis, myocardial fibrosis, neurofibromas, hypertrophic scars, various dermatological conditions, for example, atopic dermatitis and psoriasis, myocardial infarction, stroke, angina and other consequences of atherosclerotic plaque rupture, as well as periodontal disease, diabetic retinopathy, tumor growth, anaphylaxis, multiple sclerosis, peptic ulcers, and syncytial viral infections.

A special embodiment of the therapeutic methods of the present invention is the treating of asthma.

Another special embodiment of the therapeutic methods of the present invention is the treating of joint inflammation.

Another special embodiment of the therapeutic methods of the present invention is the treating of inflammatory bowel disease.

According to a further feature of the invention there is provided a method for the treatment of a human or animal patient suffering from, or subject to, conditions which can be ameliorated by the administration of an inhibitor of tryptase, for example conditions as hereinbefore described, which comprises the administration to the patient of an effective amount of compound of the invention or a composition containing a compound of the invention. "Effective amount" is meant to describe an amount of compound of the present invention effective in inhibiting tryptase and thus producing the desired therapeutic effect.

Other pharmaceutically active agents can be employed in combination with the compounds of the invention depending upon the disease being treated. For example, in the treatment of asthma, beta-adrenergic agonists such as albuterol, terbutaline, formoterol, fenoterol or prenaline can be included as can anticholinergics such as ipratropium bromide, anti-inflammatory corticosteroids such as beclomethasone dipropionate, triamcinolone acetonide, flunisolide or dexamethasone, and anti-inflammatory agents such as sodium cromoglycate and nedocromil sodium.

References herein to treatment should be understood to include prophylactic therapy as well as treatment of established conditions.

The present invention also includes within its scope pharmaceutical compositions comprising at least one of the compounds of the invention in association with a pharmaceutically acceptable carrier or excipient.

Compounds of the invention may be administered by any suitable means. In practice compounds of the present invention may generally be administered parenterally, topically, rectally, orally or by inhalation, especially by the oral route.

Compositions according to the invention may be prepared according to the customary methods, using one or more pharmaceutically acceptable adjuvants or excipients. The adjuvants comprise, inter alia, diluents, sterile aqueous media and the various non-toxic organic solvents.

The compositions may be presented in the form of tablets, pills, granules, powders, aqueous solutions or suspensions, injectable solutions, elixirs or syrups, and can contain one or more agents chosen from the group comprising sweeteners, flavourings, colourings, or stabilisers in order to obtain pharmaceutically acceptable preparations. The choice of vehicle and the content of active substance in the vehicle are generally determined in accordance with the solubility and chemical properties of the active compound, the particular mode of administration and the provisions to be observed in pharmaceutical practice. For example, excipients such as lactose, sodium citrate, calcium carbonate, dicalcium phosphate and disintegrating agents such as starch, alginic acids and certain complex silicates combined with lubricants such as magnesium stearate, sodium lauryl sulfate and talc may be used for preparing tablets. To prepare a capsule, it is advantageous to use lactose and high molecular weight polyethylene glycols. When aqueous suspensions are used they can contain emulsifying agents or agents which facilitate suspension. Diluents such as sucrose, ethanol, polyethylene glycol, propylene glycol, glycerol and chloroform or mixtures thereof may also be used.

For parenteral administration, emulsions, suspensions or solutions of the products according to the invention in vegetable oil, for example sesame oil, groundnut oil or olive oil, or aqueous-organic solutions such as water and propylene glycol, injectable organic esters such as ethyl oleate, as well as sterile aqueous solutions of the pharmaceutically acceptable salts, are used. The solutions of the salts of the products according to the invention are especially useful for administration by intramuscular or subcutaneous injection. The aqueous solutions, also comprising solutions of the salts in pure distilled water, may be used for intravenous administration with the proviso that their pH is suitably adjusted, that they are judiciously buffered and rendered isotonic with a sufficient quantity of glucose or sodium chloride and that they are sterilised by heating, irradiation or microfiltration.

For topical administration, gels (water or alcohol based), creams or ointments containing compounds of the invention may be used. Compounds of the invention may also be incorporated in a gel or matrix base for application in a patch, which would allow a controlled release of compound through the transdermal barrier.

For administration by inhalation compounds of the invention may be dissolved or suspended in a suitable carrier for use in a nebuliser or a suspension or solution aerosol, or may be absorbed or adsorbed onto a suitable solid carrier for use in a dry powder inhaler.

Solid compositions for rectal administration include suppositories formulated in accordance with known methods and containing at least one compound of the invention.

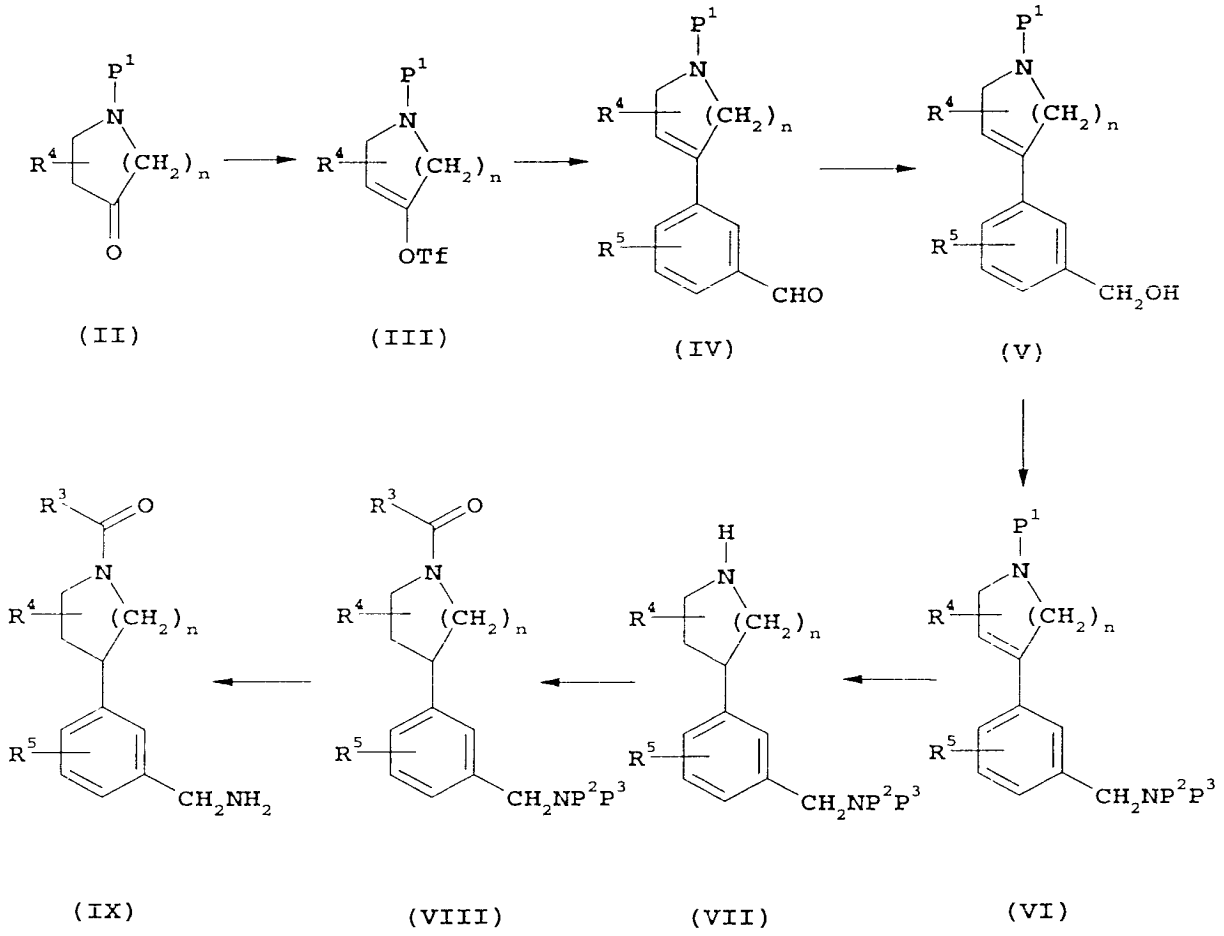
The percentage of active ingredient in the compositions of the invention may be varied, it being necessary that it should constitute a proportion such that a suitable dosage shall be obtained. Obviously, several unit dosage forms may be administered at about the same time. The dose employed will be determined by the physician, and depends upon the desired therapeutic effect, the route of administration and the duration of the treatment, and the condition of the patient. In the adult, the doses are generally from about 0.001 to about 50, preferably about 0.001 to about 5, mg/kg body weight per day by inhalation, from about 0.01 to about 100, preferably 0.1 to 70, more especially 0.5 to 10, mg/kg body weight per day by oral administration, and from about 0.001 to about 10, preferably 0.01 to 1, mg/kg body weight per day by intravenous administration. In each particular case, the doses will be determined in accordance with the factors distinctive to the subject to be treated, such as age, weight, general state of health and other characteristics which can influence the efficacy of the medicinal product.

The compounds according to the invention may be administered as frequently as necessary in order to obtain the desired therapeutic effect. Some patients may respond rapidly to a higher or lower dose and may find much weaker maintenance doses adequate. For other patients, it may be necessary to have long-term treatments at the rate of 1 to 4 doses per day, in accordance with the physiological requirements of each particular patient. Generally, the active product may be administered orally 1 to 4 times per day. Of course, for some patients, it will be necessary to prescribe not more than one or two doses per day.

Compounds of the invention may be prepared by the application or adaptation of known methods, by which is meant methods used heretofore or described in the literature, for example those described by R.C.Larock in Comprehensive Organic Transformations, VCH publishers, 1989.

In the reactions described hereinafter it may be necessary to protect reactive functional groups, for example hydroxy, amino, imino, thio or carboxy groups, where these are desired in the final product, to avoid their unwanted participation in the reactions. Conventional protecting groups may be used in accordance with standard practice, for examples see T.W. Greene and P.G.M.Wuts in "Protective Groups in Organic Chemistry" John Wiley and Sons, 1991.

Compounds of formula (I) wherein R^3, R^4, R^5 and n are as hereinbefore defined, R^1 and R^2 are both hydrogen and ------ is a single bond, represented by formula (IX), may be prepared as shown in scheme 1.

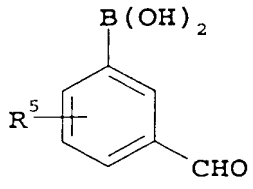


Scheme 1

For example compounds of formula (IX) may be prepared by:-

(i) treating compounds of formula (II) wherein R^4 and n are as hereinbefore defined and P^1 is a suitable protecting group, such as benzyloxycarbonyl, with a suitable base, such as lithium hexamethyldisilazane, in an inert solvent, such as tetrahydrofuran, and at a temperature at about -78°C, followed by reaction with N-phenyltrifluoromethanesulfonimide to give compounds of formula (III) wherein R^4, P^1 and n are as hereinbefore defined and Tf is $-SO_2CF_3$;

(ii) reaction of triflates of formula (III) with an aryl boronic acid of formula (X):-



(X)

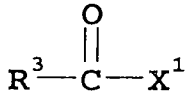
in the presence of an aqueous base such as sodium bicarbonate and a palladium catalyst such as palladium tetrakistriphenylphosphine, and at a temperature from about 80 to about 100°C, to give compounds of formula (IV) wherein R^4, R^5, P^1 and n are as hereinbefore defined;

(iii) reduction of compounds of formula (IV) with sodium borohydride in ethanol to give compounds of formula(V) wherein R^4, R^5, P^1 and are as hereinbefore defined;

(iv) conversion of the hydroxymethyl group in compounds of formula (V) to an aminomethyl group which is suitably protected to facilitate the further processes described hereinafter - for example reaction of compounds of formula (V) with phosphorus tribromide in pyridine followed by treatment of the resultant bromomethyl intermediate with di-tert-butyliminodicarboxylate to give compounds of formula (VI) wherein R^4, R^5, P^1 and n are as hereinbefore defined and P^2 and P^3 are each tertiary-butyloxycarbonyl (a suitable protecting group that is stable under conditions for the subsequent removal of protecting group P^1);

(v) removal of the protecting group P^1 in compounds of formula (VI), for example when P^1 is benzyloxycarbonyl and P^2 and P^3 are both tertiary-butyloxycarbonyl, the deprotection may conveniently be carried out by hydrogenation in the presence of a suitable metal catalyst, e.g. platinum or palladium optionally supported on an inert carrier such as carbon, preferably in a solvent such as methanol or ethanol to give compounds of formula (VII) wherein R^4, R^5 and n are as hereinbefore defined and P^2 and P^3 are as just defined;

(vi) reaction of compounds of formula (VII) with compounds of formula (XI):-

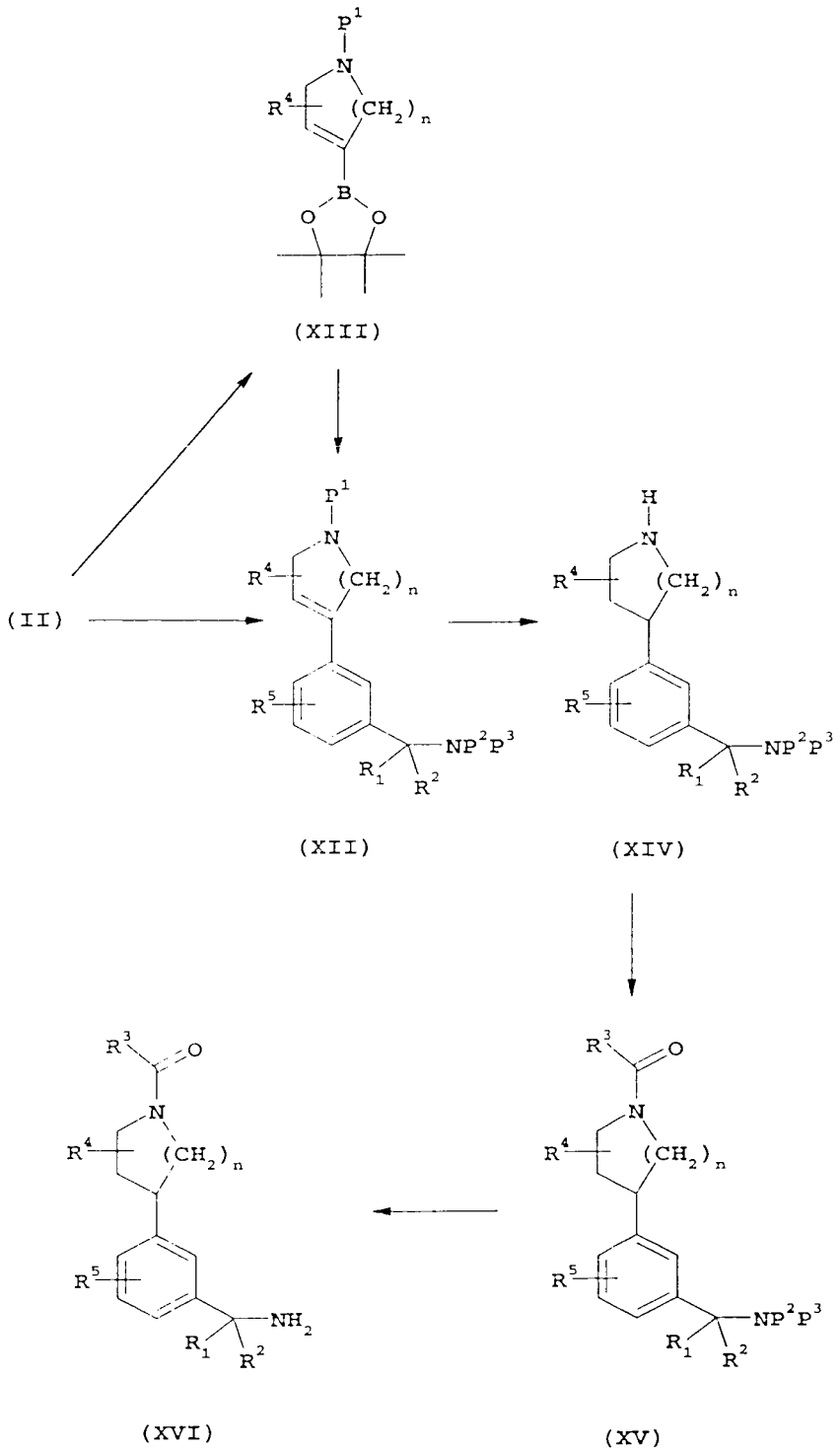


(XI)

wherein R^3 is as hereinbefore defined and X^1 is a hydroxy group, or a halogen, preferably chlorine, atom using standard coupling conditions [for example when X^1 is a hydroxy group the reaction may be carried out using standard peptide coupling procedures for example coupling in the presence of O-(7-azabenzotriazol-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate and triethylamine (or diisopropylethylamine) in tetrahydrofuran (or dimethylformamide), at room temperature; and when X^1 is a halogen atom the acylation reaction may be carried out with the aid of a base, such pyridine, preferably in a solvent such as tetrahydrofuran and at a temperature at about room temperature] to give compounds of formula (VIII) wherein R^3, R^4, R^5, n, P^2 and P^3 are as defined above;

(vii) removal of the protecting groups P^2 and P^3 in compounds of formula (VIII), for example when P^2 and P^3 are both tertiary-butyloxycarbonyl the reaction may conveniently be carried out in the presence of an acid such as trifluoracetic acid in an inert solvent, such as dichloromethane, or by treatment with hydrogen chloride in methanol.

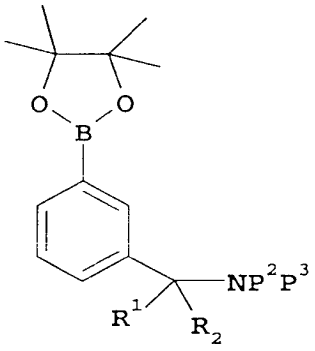
Compounds of formula (I) wherein R^1, R^2, R^3, R^4, R^5 and n are as hereinbefore defined and ----- is a single bond, represented by formula (XVI), may be prepared as shown in scheme 2.



Scheme 2

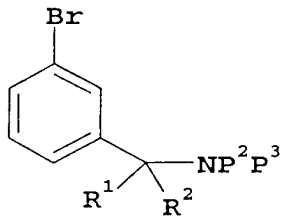
For example compounds of formula (XVI) may be prepared by:-

(i) reaction of compounds of formula (II) wherein R^4 and n are as hereinbefore defined and P^1 is a suitable protecting group, such as benzyloxycarbonyl, with an aryl boronate of formula (XVII):-



(XVII)

wherein R^1, R^2, P^2 and P^3 are as hereinbefore defined in the presence of potassium carbonate and a palladium catalyst such as [1,1'-bis-(diphenylphosphino)ferroceno]-dichloropalladium (II)-dichloromethane complex in an inert solvent, such as dimethylsulfoxide, and at a temperature at about 80°C to give compounds of formula (XII) wherein R^1, R^2, R^4, R^5, n, P^1, P^2 and P^3 are as hereinbefore defined; alternatively compounds of formula (XII) may be prepared by reaction of compounds of formula (II) with bis(pinacolato)boron in the presence of potassium acetate, (diphenylphosphino)-ferrocene and [1,1'-bis-(diphenylphosphino)ferroceno]-dichloropalladium (II), in an inert solvent, such as dioxane, and at a temperature at about 80°C followed by reaction of the intermediate boronate of formula (XIII) with compounds of formula (XVIII):-



(XVIII)

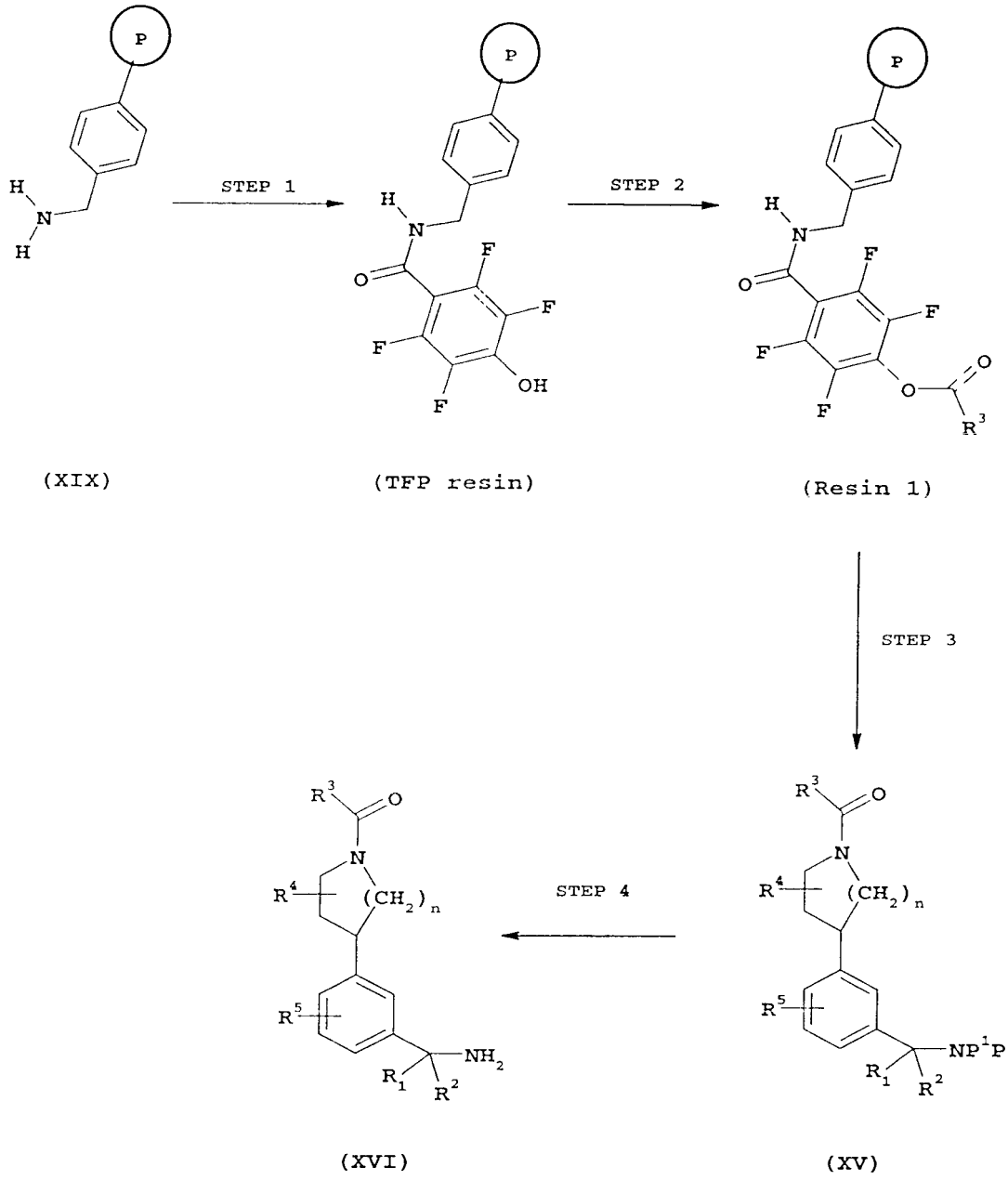
wherein R^1, R^2, P^1 and P^2 are as hereinbefore defined in the presence of potassium carbonate and a palladium catalyst such as [1,1'-bis-(diphenylphosphino)ferroceno]-dichloropalladium (II)-dichloromethane complex in an inert solvent, such as dimethylsulfoxide, and at a temperature at about 80°C];

(ii) removal of the protecting group P^1 in compounds of formula (XII), for example when P^1 is benzyloxycarbonyl and P^2 and P^3 are both tertiary-butyloxycarbonyl, the deprotection may conveniently be carried out by hydrogenation as described hereinabove to give compounds of formula (XIV) wherein R^1, R^2, R^4, R^5, n, P^2 and P^3 are as just defined;

(iii) reaction of compounds of formula (XIV) with compounds of formula (XI) wherein R^1 is as hereinbefore defined and X^1 is a hydroxy group, or a halogen, preferably chlorine, atom using standard coupling conditions [for example those described hereinabove] to give compounds of formula (XV) wherein R^1, R^2, R^3, R^4, R^5, n, P^2 and P^3 are as defined above;

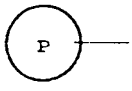
(vii) removal of the protecting groups P^2 and P^3 in compounds of formula (XV), using standard coupling conditions [for example those described hereinabove].

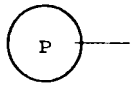
Compounds of formula (I) wherein R^1, R^2, R^3, R^4, R^5 and n are as hereinbefore defined and ------ is a single bond, represented by formula (XVI), may also be prepared using resin technology as shown in scheme 3:-

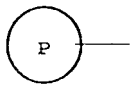


Scheme 3

For example compounds of formula (XVI) may be prepared by:-

(i) Coupling of the resin (XIX, an aminomethylated styrene/divinylbenzene copolymer), where  represents the polymeric core (comprising polystyrene crosslinked with 1% to 2% divinylbenzene), with 4-hydroxy-2,3,5,6-tetrafluorobenzoic acid, according to the procedure described by J.M.Salvino et. al. in International Patent

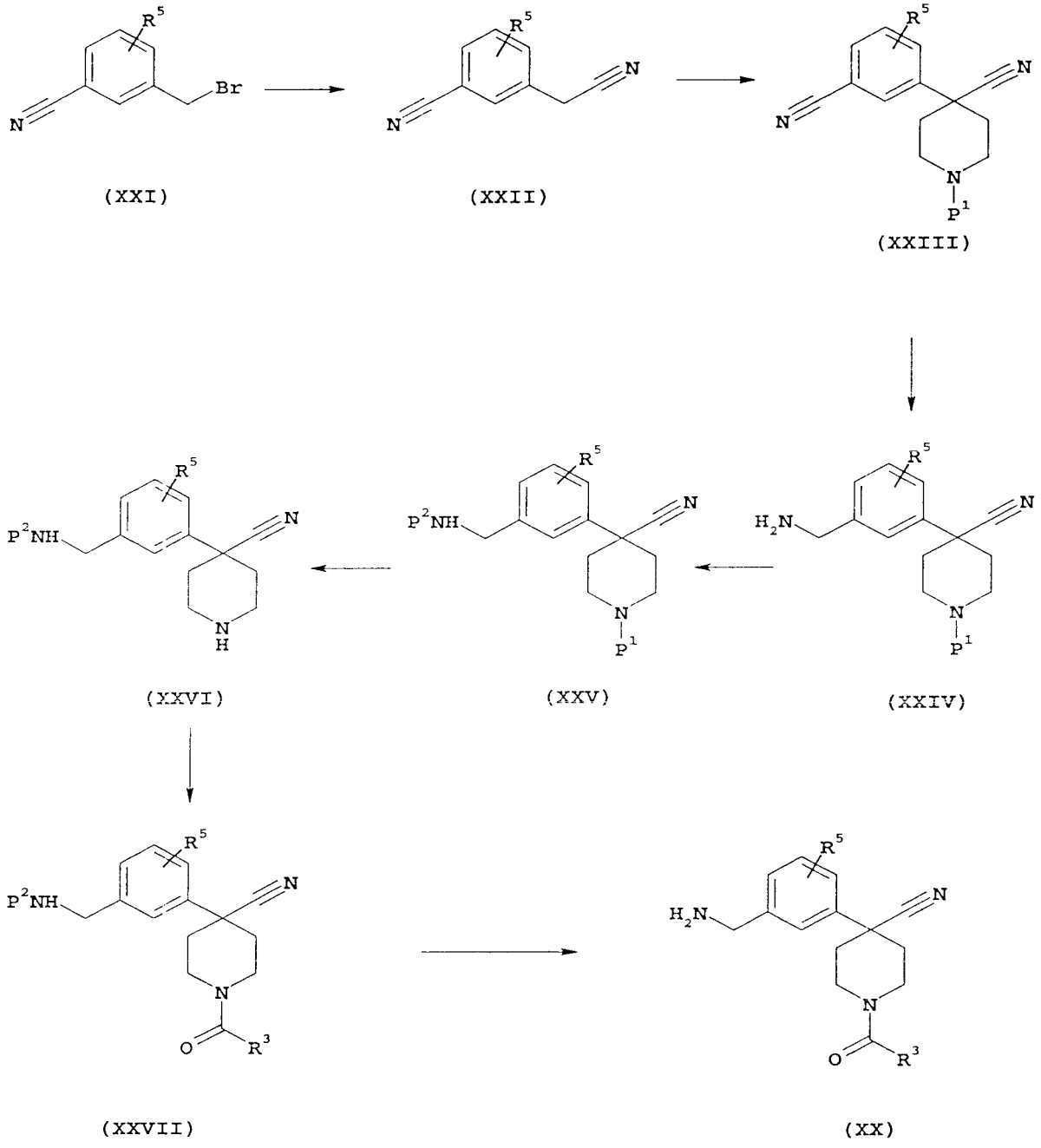
Application Publication No. WO 99/67228, to give TFP resin wherein  is as hereinbefore defined;

(ii) treatment of TFP resin with acids of formula (XI) wherein R^3 is as hereinbefore defined and X^1 is hydroxy, in the presence of diisopropyl carbodiimide and dimethylaminopyridine, in an inert solvent, such as dimethylformamide, and at a temperature at about room temperature, to give resin 1 wherein R^3 and  are hereinbefore defined:

(iii) treatment of resin 1 with compounds of formula (XIV) wherein R^1, R^2, R^4, R^5, n, P^1 and P^2 are as defined immediately hereinbefore, in an inert solvent, such as dichloromethane, and at a temperature at about room temperature, to give compounds of formula (XV);

(iv) removal of the protecting groups in compounds of formula (XV), for example when P^2 and P^3 are both tertiary-butyloxycarbonyl the reaction may conveniently be carried out in the presence of an acid such as trifluoracetic acid in an inert solvent, such as dichloromethane, or by treatment with hydrogen chloride in methanol.

Compounds of formula (Ia), wherein R^3 and R^5 are as hereinbefore defined and R^4 is cyano attached at the 4 position of the piperidine ring, represented by formula (XX), may be prepared as shown in scheme 4:-

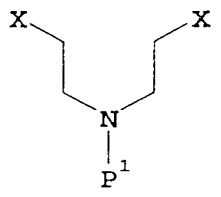


Scheme 4

For example compounds of formula (XX) may be prepared by:-

(i) Reacting a benzyl bromide of formula (XXI) wherein R^5 is as hereinbefore defined, with sodium cyanide, in the presence of a phase transfer catalyst, such as tetrabutylammonium bromide, in a mixture of water and an inert solvent, such as dichloromethane, and at a temperature at about room temperature to give compounds of formula (XXII) wherein R^5 is as hereinbefore defined;

(ii) treatment of the benzylcyanides of formula (XXII) with a suitably protected bis-(2-haloethyl)amine of formula (XXVIII):-



(XXVIII)

wherein X is halo, preferably chloro, and P^1 is a suitable protecting group, such as tertiary-butyloxycarbonyl, in the presence of sodium hydride and in an inert solvent, such as dimethylformamide, and at a temperature at about room temperature, to give compounds of formula (XXIII) wherein R^5 and P^1 are as hereinbefore defined;

(iii) hydrogenation of compounds of formula (XXIII) in the presence of hydrochloric acid in ethanol and under pressure to give compounds of formula (XXIV) wherein R^5 and P^1 are as hereinbefore defined;

(iv) protection of the amino group in compounds of formula (XXIV) with a suitable protecting group, for example with a benzyloxycarbonyl group, to give compounds of formula (XXV) wherein R^5, P^1 and P^2 are as hereinbefore defined;

(v) removal of the protecting group P^1 in compounds of formula (XXV) to give compounds of formula (XXVI) wherein R^5 and P^2 are as hereinbefore defined;

(vi) reaction of compounds of formula (XXVI) with compounds of formula (XI) wherein R^1 is as hereinbefore defined and X^1 is a hydroxy group, or a halogen, preferably chlorine, atom using standard coupling conditions [for example those described hereinabove] to give compounds of formula (XXVII) wherein R^5 and P^2 are as defined above;

(vii) removal of the protecting groups P^2 in compounds of formula (XXVII), using standard coupling conditions [for example those described hereinabove].

Compounds of formula (I) wherein R^3, R^4, R^5 and n are as hereinbefore defined, R^1 and R^2 are both hydrogen and ⋯⋯ is a double bond, represented by formula (IX), may be prepared by removal of the P^1 protecting group in compounds of formula (VI) followed by acylation with compounds of formula (XI) and subsequent removal of the P^2 and P^3 protecting groups.

Compounds of formula (I) wherein R^1, R^2, R^3, R^4, R^5 and n are as hereinbefore defined and ⋯⋯ is a double bond, represented by formula (XVI), may be prepared by removal of the P^1 protecting group in compounds of formula (XII) followed by acylation with compounds of formula (XI) and subsequent removal of the P^2 and P^3 protecting groups.

According to a further feature of the present invention, compounds of the invention may be prepared by interconversion of other compounds of the invention.

As an example of the interconversion process, compounds of formula (I) wherein R^1, R^2, R^4, R^5 and n are as hereinbefore defined and R^3 contains an optionally substituted alkylene linkage, may be prepared by hydrogenation of the corresponding compounds of formula (I) in which R^3 contains the corresponding optionally substituted alkenylene or alkynylene linkage. The hydrogenation may be carried out using hydrogen (optionally under pressure) in the presence of a suitable metal catalyst, e.g. platinum or palladium optionally supported on an inert carrier such as carbon, preferably in a solvent such as methanol or ethanol, and at a temperature at about room temperature.

As another example of the interconversion process, compounds of the invention containing a heterocyclic group wherein the hetero atom is a nitrogen atom may be oxidised to their corresponding N-oxides. The oxidation may conveniently be carried out by means of reaction with a mixture of hydrogen peroxide and an organic acid, e.g. acetic acid, preferably at or above room temperature, for example at a temperature of about 60-90°C. Alternatively, the oxidation may be carried out by reaction with a peracid, for example peracetic acid or m-chloroperoxybenzoic acid, in an inert solvent such as chloroform or dichloromethane, at a temperature from about room temperature to reflux, preferably at elevated temperature. The oxidation may alternatively be carried out by reaction with hydrogen peroxide in the presence of sodium tungstate at temperatures between room temperature and about 60°C.

As another example of the interconversion process, compounds of formula (I) containing sulfone linkages may be prepared by the oxidation of corresponding compounds containing -S- or sulfoxide linkages. For example, the oxidation may conveniently be carried out by means of reaction with a peroxyacid, e.g. 3-chloroperbenzoic acid, preferably in an inert solvent, e.g. dichloromethane, preferably at or near room temperature.

It will be appreciated that compounds of the present invention may contain asymmetric centres. These asymmetric centres may independently be in either the R or S configuration. It will be apparent to those skilled in the art that certain compounds of the invention may also exhibit geometrical isomerism. It is to be understood that the present invention includes individual geometrical isomers and stereoisomers and mixtures thereof, including racemic mixtures, of compounds of formula (I) hereinabove. Such isomers can be separated from their mixtures, by the application or adaptation of known methods, for example chromatographic techniques and recrystallisation techniques, or they are separately prepared from the appropriate isomers of their intermediates.

According to a further feature of the invention, acid addition salts of the compounds of this invention may be prepared by reaction of the free base with the appropriate acid, by the application or adaptation of known methods. For example, the acid addition salts of the compounds of this invention may be prepared either by dissolving the free base in water or aqueous alcohol solution or other suitable solvents containing the appropriate acid and isolating the salt by evaporating the solution, or by reacting the free base and acid in an organic solvent, in which case the salt separates directly or can be obtained by concentration of the solution.

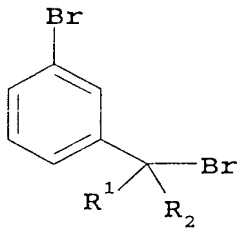
The acid addition salts of the compounds of this invention can be regenerated from the salts by the application or adaptation of known methods. For example, parent compounds of the invention can be regenerated from their acid addition salts by treatment with an alkali, e.g. aqueous sodium bicarbonate solution or aqueous ammonia solution.

The starting materials and intermediates may be prepared by the application or adaptation of known methods, for example methods as described in the Reference Examples or their obvious chemical equivalents.

Aryl boronates of formula (XVII) wherein R^1, R^2, P^2 and P^3 are as hereinbefore defined may be prepared by reaction of compounds of formula (XVIII) wherein R^1, R^2, P^2 and P^3 are as

hereinbefore defined, with bis(pinacolato)boron in the presence of potassium acetate and [1,1'-bis-(diphenylphosphino)ferroceno]-dichloropalladium (II) in an inert solvent, such as dioxane, at and at a temperature at about 80°C.

Compounds of formula (XVIII) wherein R^1 and R^2 are as hereinbefore defined and P^2 and P^3 are both tertiary-butoxycarbonyl may be prepared by reaction of compounds of formula (XXIX):-



(XXIX)

wherein R^1 and R^2 are as hereinbefore defined with sodium hydride and di-tertiary-butyliminodicarboxylate, in an inert solvent, such as tetrahydrofuran, and at a temperature at about room temperature.

Intermediates of formulae (VIII) and (XV) are novel compounds and, as such, they and their processes described herein for their preparation constitute further features of the present invention.

The present invention is further Exemplified but not limited by the following illustrative Examples and Reference Examples.

In the nuclear magnetic resonance spectra (NMR) the chemical shifts are expressed in ppm relative to tetramethylsilane. Abbreviations have the following significances: br = broad, dd = double doublet, s = singlet; m = multiplet.

High Pressure Liquid Chromatography/ Mass Spectrometry (LC-MS) conditions for determination of retention times (R_T) were as follows: 3 micron Luna C18 (2) HPLC column (30mm x 4.6mm) eluting with (i) mixture of 0.05% trifluoroacetic acid in acetonitrile and 0.05% trifluoroacetic acid in water (1:19, v/v) for 2 minutes, (ii) mixture of 0.05% trifluoroacetic acid in acetonitrile and 0.05% trifluoroacetic acid in water (1:19 to 19:1, v/v) gradient elution over 10 minutes, (iii) mixture of 0.05% trifluoroacetic acid in acetonitrile and 0.05% trifluoroacetic acid

in water (19:1, v/v) for 2 minutes, (iv) mixture of 0.05% trifluoroacetic acid in acetonitrile and 0.05% trifluoroacetic acid in water (1:19 to 1:19, v/v) gradient elution over 2 minutes; flow rate 2ml/minute with approximately 200µl/minute split to the Mass Spectrometer; injection volume 10-40µl; in line Diode Array (220-450nm), in line Evaporative light scattering (ELS) detection ELS - temperature 50°C, Gain 8 - 1.8ml/minute; Source temperature 150°C.

EXAMPLE 1

3-[1-(5-Phenylethynyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride

A. *B*-{3-[*N*,*N*-bis-(*tert*-butoxycarbonyl)aminomethyl]-phenyl}-pinacolato-boron

A solution of 3-bromobenzylbromide (7.5g, 30mmol) and di-*tert*-butyliminodicarboxylate (6.5g, 30mmol) in anhydrous tetrahydrofuran (80ml) was treated portionwise with sodium hydride (1.2g, 60% dispersion in mineral oil). After stirring at ambient temperature for 7 hours the reaction mixture was partitioned between saturated aqueous ammonium chloride solution (90ml) and ethyl acetate (2 lots of 250ml). The combined organic layers were washed with brine (75ml), then dried over magnesium sulfate and then concentrated under vacuum. The residue was subjected to chromatography on silica gel eluting with a mixture of pentane and dichloromethane (2:1, v/v) to give 3-[*N*,*N*-bis-(*tert*-butoxycarbonyl)aminomethyl]-bromobenzene as a pale yellow oil (9.52g). A sample of this material (2.0g, 5.2mmol) was dissolved in anhydrous dimethylsulfoxide (30ml) and the solution was treated with potassium acetate (1.52g, 15.5mmol), bis(pinacolato)diboron (1.45g, 5.7mmol), and [1,1'-bis-(diphenylphosphino)ferroceno]-dichloropalladium (II)-dichloromethane complex (0.13g, 0.16mmol). This mixture was stirred at 80°C under an atmosphere of nitrogen for 5 hours, then cooled and then partitioned between water (100ml) and diethyl ether (4 lots of 75ml). The combined organic layers were washed twice with brine (75ml), then dried over magnesium sulfate and then concentrated under vacuum. The residue was subjected to chromatography on silica gel eluting with a mixture of pentane and dichloromethane (2:1, v/v) to give *B*-{3-[*N*,*N*-bis-(*tert*-butoxycarbonyl)-aminomethyl]-phenyl}-pinacolato-boron as a colourless oil (1.08g). ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.78 (s, 1H), 7.70 (m, 1H), 7.39 (m, 1H), 7.30 (m, 1H), 4.79 (s, 2H), 1.27 (s, 18H), 1.35 (s, 12H). MS(EI): 434(M^++H).

B. 4-{3-[*N*,*N*-Bis-(*tert*-butoxycarbonyl)aminomethyl]-phenyl}-piperidine

A solution of lithium diisopropylamine (54mmol) in anhydrous tetrahydrofuran (50ml), at -78°C, was treated dropwise with a solution of benzyl 4-oxo-1-piperidinecarboxylate (11.4g, 49mmol) in anhydrous tetrahydrofuran (50ml). This mixture was stirred at -78°C for 20 minutes and then

treated with a solution of N-phenyltrifluoromethanesulfonimide (19.26g, 54mmol) in anhydrous tetrahydrofuran (55ml). The resultant orange suspension was warmed to 0°C, then stirred at 0°C for 2 hours and then concentrated under vacuum. The residue was subjected to chromatography on silica gel eluting with dichloromethane to yield benzyl 1,2,3,6-tetrahydro-4-(trifluoromethylsulfonyloxy)-pyridine-1-carboxylate as a yellow oil (11.34g). A portion of this material (0.84g, 2.3mmol) was dissolved in anhydrous dimethylformamide (20ml) and the solution was treated with *B*-{3-[*N,N*-bis-(*tert*-butoxycarbonyl)aminomethyl]-phenyl-pinacolato-boron (1.0g, 2.3mmol), potassium carbonate (0.96g, 6.7mmol) and [1,1'-bis-(diphenylphosphino)ferroceno]dichloropalladium (II)-dichloromethane complex (0.1g, 0.14mmol). This mixture was heated at 80°C under an atmosphere of nitrogen for 18 hours, then cooled and then concentrated under vacuum. The residue was partitioned between ethyl acetate (2 lots of 100ml) and water (100ml) containing concentrated ammonium hydroxide (6ml). The combined organic extracts were dried over magnesium sulfate and then concentrated under vacuum. The resultant oil was subjected to chromatography on silica gel eluting with a mixture of ethyl acetate and pentane (1:4, v/v) to yield a yellow oil (0.9g). This material was dissolved in ethanol (20ml) and the solution was treated with 10% palladium on carbon (20 mg) then stirred at ambient temperature under an atmosphere of hydrogen for 5 hours. The reaction mixture was filtered through a short pad of hyflo and the filtrate was concentrated under vacuum to give 4-{3-[*N,N*-bis-(*tert*-butoxycarbonyl)aminomethyl]-phenyl}-piperidine as colourless oil (0.54g). ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.10 (m, 4H), 4.80 (s, 2H), 4.45 (br m,1H), 3.20 (br m, 1H), 2.98 (br m, 1H), 2.75 (br m, 1H), 1.90 (m, 1H), 1.75-1.60 (m, 3H), 1.42 (s, 18H). MS(EI): 391(M^++H).

C. *N,N*-Bis-(*tert*-butoxycarbonyl)-3-[1-(5-phenylethynyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine

A solution of 5-phenylethynyl-pyridine-3-carboxylic acid (0.25g, 1.1mmol) in anhydrous dimethylformamide (5ml) was treated with O-(7-azabenzotriazol-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate (0.42g, 1.1mmol) and diisopropylethylamine (0.5ml, 3mmol). This mixture was stirred 15 minutes at ambient temperature and then treated with a solution of 4-{3-[*N,N*-Bis-(tert-butoxycarbonyl)aminomethyl]-phenyl}-piperidine (0.39g, 1.0mmol) in dimethylformamide (5ml). After stirring at ambient temperature for 18 hours the reaction mixture was concentrated under vacuum. The residue was partitioned between ethyl acetate (50ml) and saturated aqueous sodium bicarbonate (15ml). The organic layer was dried over magnesium sulfate and then concentrated under vacuum. The residue was subjected to chromatography on silica gel eluting with a mixture of dichloromethane and methanol (49:1, v/v) to give *N,N*-bis-(*tert*-butoxycarbonyl)-3-[1-(5-phenylethynyl-pyridine-3-carbonyl)-piperidin-4-yl]-

benzylamine as a yellow oil (0.25g). ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.82 (s, 1H), 8.62 (s, 1H), 8.02 (s, 1H), 7.61 (m, 2H), 7.45 (m, 3H), 7.30 (m, 1H), 7.20 (m, 1H), 7.15 (m, 1H), 7.04 (m, 1H), 4.68 (s, 2H), 4.62 (br m, 1H), 3.60 (br m, 1H), 3.23 (br m, 1H), 2.85 (m, 2H), 1.83 (br m, 1H), 1.65 (m, 3H), 1.39 (s, 18H). MS(EI): 596(M^++H).

D. 3-[1-(5-Phenylethynyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride

A solution of 3-[1-(5-phenylethynyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzonitrile (0.15g, 0.25mmol) in ethyl acetate (20ml) was cooled to 0°C and then saturated with hydrogen chloride gas. The reaction mixture was stirred at ambient temperature for 4 hours and then concentrated to dryness under vacuum. The residue was treated with ethyl acetate (10ml) and the solvent removed under vacuum. This process was repeated twice to give 3-[1-(5-phenylethynyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride as a white solid (0.11g). ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.83 (s, 1H), 8.63 (s, 1H), 8.08 (s, 1H), 7.61 (m, 2H), 7.45 (m, 3H), 7.44 (s, 1H), 7.37 (m, 3H), 4.64 (br m, 1H), 4.00 (m, 2H), 3.62 (br m, 1H), 3.25 (br m, 1H), 2.90 (br m, 2H), 1.88 (br m, 1H), 1.70 (m, 3H). MS(EI): 396(M^++H).

EXAMPLE 2

3-[1-(5-Phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride

A. 5-Phenylethyl-pyridine-3-carboxylic acid

A solution of 5-phenylethynyl-pyridine-3-carboxylic acid (2g, 8.9mmol) in tetrahydrofuran (50ml) was treated with 10% palladium on carbon (200 mg) and stirred at ambient temperature under an atmosphere of hydrogen for 5 hours. The reaction mixture was filtered through a short pad of hyflo and the filtrate was concentrated under vacuum to give 5-phenylethyl-pyridine-3-carboxylic acid as white solid (2g). ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.90 (m, 1H), 8.60 (m, 1H), 8.12 (m, 1H), 7.21 (m, 5H), 3.38 (br s, 1H), 2.95 (m, 4H).

B. *N,N*-Bis-(*tert*-butoxycarbonyl)-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine

By proceeding in a similar manner to the method described in EXAMPLE 1C, but using of 5-phenylethyl-pyridine-3-carboxylic acid in place of 5-phenylethynyl-pyridine-3-carboxylic acid, there was prepared *N,N*-bis-(*tert*-butoxycarbonyl)-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.50 (s, 1H), 8.41 (s, 1H), 7.61 (m, 1H), 7.30-7.05 (m, 9H), 4.68 (s, 2H), 4.62 (br m, 1H), 3.48 (br m,

1H), 3.35 (s, 4H), 3.18 (br m, 1H), 2.85 (m, 2H), 1.82 (br m, 1H), 1.65 (br m, 1H), 1.58 (br m, 2H), 1.39 (s, 18H). MS(EI): 600(M^++H).

C. 3-[1-(5-Phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 1D, but using *N,N*-bis-(*tert*-butoxycarbonyl)-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine in place of *N,N*-Bis-(tert-butoxycarbonyl)-3-[1-(5-phenylethynyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine, there was prepared 3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride as a white solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.90 (m, 2H), 8.42 (s, 1H), 7.58 (m, 1H), 7.38-7.20 (m, 8H), 4.62 (br m, 1H), 4.00 (m, 2H), 3.45 (br m, 1H), 3.20 (br m, 1H), 3.17 (m, 2H), 3.00 (m, 2H), 2.90 (br m, 2H), 1.88 (br m, 1H), 1.70 (br m, 3H). MS(EI): 400(M^++H).

EXAMPLE 3

3-[1-(1-oxy-5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine hydrochloride

A solution of *N,N*-Bis-(*tert*-butoxycarbonyl)-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine (100mg, 0.17mmol, EXAMPLE 2B) in dichloromethane (10ml) was treated with meta-chloroperbenzoic acid (80%, 80mg, 0.37mmol). After stirring for 18 hours at ambient temperature the reaction mixture was diluted with dichloromethane (40ml) and then washed three times with saturated aqueous sodium bicarbonate solution (20ml). The organic phase was dried over magnesium sulfate and then concentrated under vacuum. The residue was subjected to chromatography on silica gel eluting with a mixture of dichloromethane and methanol (98:2, v/v) to give *N,N*-bis-(*tert*-butoxycarbonyl)-3-[1-(1-oxy-5-phenylethyl-pyridine-3-carbonyl)-piperidin-4 yl]-benzylamine as a colourless oil (70mg). This material was dissolved in ethyl acetate (10ml) and the solution was cooled to 0°C, then saturated with hydrogen chloride gas, then stirred at ambient temperature for 4 hours and then concentrated to dryness under vacuum. The residue was treated with ethyl acetate (10ml) and the solvent removed under vacuum. This process was repeated twice to leave the title compound as a white solid (45mg). ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.39 (s, 1H), 8.30 (s, 1H), 7.45 (s, 1H), 7.40 (s, 1H), 7.38-7.18 (m, 8H), 4.59 (br m, 1H), 4.00 (m, 2H), 3.50 (br m, 1H), 3.20 (br m, 1H), 2.98 (s, 4H), 2.85 (br m, 2H), 1.84 (br m, 1H), 1.68 (br m, 3H). MS(EI): 416(M^++H).

EXAMPLE 4

3-[1-(Quinoline-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 1, but using quinoline-3-carboxylic acid in place of 5-phenylethynyl-pyridine-3-carboxylic acid, there was prepared the title compound as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 9.10 (s, 1H), 8.78 (s, 1H), 8.20 (m, 2H), 7.98 (m, 1H), 7.80 (m, 1H), 7.50 (s, 1H), 7.35 (m, 3H), 4.72 (br m, 1H), 4.00 (m, 2H), 3.80 (br m, 1H), 3.30 (br m, 1H), 2.90 (br m, 2H), 1.90 (br m, 1H), 1.75 (br m, 3H). MS(EI): 346(M^++H).

EXAMPLE 5

3-[1-(3-Phenylethynyl-benzoyl)-piperidin-4-yl]-benzylamine hydrochloride

A. *N,N*-Bis-(*tert*-butoxycarbonyl)-3-[1-(3-ethynyl-benzoyl)-piperidin-4-yl]-benzylamine

By proceeding in a similar manner to the method described in EXAMPLE 1C, but using of 3-ethynyl-benzoic acid [prepared according to the procedure described by C. Eaborn et al., J. Chem. Soc. C, 1967, (15), pages 1364-1366] in place of 5-phenylethynyl-pyridine-3-carboxylic acid, there was prepared *N,N*-bis-(*tert*-butoxycarbonyl)-3-[1-(3-ethynyl-benzoyl)-piperidin-4-yl]-benzylamine as a white amorphous solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.76 (m, 2H), 7.40 (m, 2H), 7.25 (m, 2H), 7.10 (m, 4H), 4.95 (br m,1H), 4.79 (s, 2H), 3.80 (br m, 1H), 3.10 (br m, 1H), 2.84 (br m, 1H), 2.78 (br m, 1H), 1.90 (m, 1H), 1.75 (m, 3H), 1.42 (s, 18H). MS(EI): 541(M^++Na).

B. *N,N*-Bis-(*tert*-butoxycarbonyl)-3-[1-(3-phenylethynyl-benzoyl)-piperidin-4-yl]-benzylamine

A mixture of *N,N*-bis-(*tert*-butoxycarbonyl)-3-[1-(3-ethynyl-benzoyl)-piperidin-4-yl]-benzylamine (0.24g, 0.46mmol), iodobenzene (95mg, 0.46mmol), dichlorobis(triphenylphosphine)palladium (II) (35mg, 0.05mmol), copper (I) iodide (26mg, 0.14mmol), triethylamine (0.57ml, 4.1mmol) and anhydrous dimethylformamide (8ml) was stirred at ambient temperature under nitrogen for 18 hours. The solvent was removed under vacuum and the residue was partitioned between ethyl acetate (3 lots of 50ml) and water (20ml). The combined organic layers were washed with brine (50ml), then dried over magnesium sulfate and then concentrated under vacuum. The residue was subjected to chromatography on silica gel eluting with a mixture of cyclohexane and ethyl acetate (3:2, v/v) to give *N,N*-bis-(*tert*-butoxycarbonyl)-3-[1-(3-phenylethynyl-benzoyl)-piperidin-4-yl]-benzylamine as a yellow oil (0.23g). ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.58 (m, 4H), 7.40 (m, 5H), 7.25 (m, 1H), 7.10 (m, 3H), 4.90 (br m,1H), 4.79 (s, 2H), 3.80 (br m, 1H), 3.15 (br m, 1H), 2.84 (br m, 1H), 2.78 (br m, 1H), 1.95 (m, 1H), 1.80 (m, 3H), 1.42 (s, 18H). MS(EI): 617(M^++ Na).

C. 3-[1-(3-Phenylethynyl-benzoyl)-piperidin-4-yl]-benzylamine hydrochloride

A solution of *N,N*-Bis-(*tert*-butoxycarbonyl)-3-[1-(3-phenylethynyl-benzoyl)-piperidin-4-yl]-benzylamine (100mg, 0.17mmol) in methanol (10ml), cooled to 0 °C, was saturated with hydrogen chloride gas. The mixture was stirred at ambient temperature for 4 hours then concentrated to dryness under vacuum. The residue was triturated with a mixture of dichloromethane and diethyl ether to give 3-[1-(3-phenylethynyl-benzoyl)-piperidin-4-yl]-benzylamine hydrochloride as a white amorphous solid (46mg). ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 7.61-7.30 (m, 13H), 4.62 (br m, 1H), 4.00 (s, 2H), 3.62 (br m, 1H), 3.25 (br m, 1H), 2.85 (br m, 2H), 1.88 (br m, 1H), 1.70 (br m, 3H). MS(EI): 395(M^++H).

EXAMPLE 6

2-{3-[4-(3-Aminomethyl-phenyl)-piperidine-1-carbonyl]-phenyl}-1-(4-hydroxyphenyl)-ethanone

A. *N,N*-Bis-(*tert*-butoxycarbonyl)-3-{1-[3-(4-hydroxyphenyl)ethynyl-benzoyl]-piperidin-4-yl}-benzylamine

By proceeding in a similar manner to the method described in EXAMPLE 5B, but using 4-iodophenol in place of iodobenzene, there was prepared *N,N*-bis-(*tert*-butoxycarbonyl)-3-{1-[3-(4-hydroxyphenyl)ethynyl-benzoyl]-piperidin-4-yl}-benzylamine as a yellow amorphous solid. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.58 (m, 2H), 7.40 (m, 4H), 7.25 (m, 1H), 7.15 (m, 3H), 6.80 (m, 2H), 4.90 (br m,1H), 4.79 (s, 2H), 3.90 (br m, 1H), 3.15 (br m, 1H), 2.84 (br m, 1H), 2.78 (br m, 1H), 1.98 (m, 1H), 1.80 (m, 3H), 1.42 (s, 18H). MS(EI): 633(M^++ Na).

B. 2-{3-[4-(3-Aminomethyl-phenyl)-piperidine-1-carbonyl]-phenyl}-1-(4-hydroxyphenyl)-ethanone hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 5C but using *N,N*-bis-(*tert*-butoxycarbonyl)-3-{1-[3-(4-hydroxyphenyl)ethynyl-benzoyl]-piperidin-4-yl}-benzylamine, there was prepared 2-{3-[4-(3-aminomethyl-phenyl)-piperidine-1-carbonyl]-phenyl}-1-(4-hydroxyphenyl)-ethanone hydrochloride as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 7.92 (m, 2H), 7.40-7.28 (m, 8H), 6.82 (m, 2H), 4.60 (br m, 1H), 4.38 (s, 2H), 4.00 (s, 2H), 3.62 (br m, 1H), 3.18 (br m, 1H), 2.80 (br m, 2H), 1.88 (br m, 1H), 1.70-1.60 (br m, 3H), MS(EI): 429(M^++H).

EXAMPLE 7

3-{1-[3-(6-Amino-pyridin-3-yl)ethynyl-benzoyl]-piperidin-4-yl}-benzylamine hydrochloride

A. 3-Iodo-6-(*tert*-butoxycarbonylamino)pyridine

A stirred solution of 6-amino-3-iodopyridine (0.44g, 2.0mmol) in anhydrous tetrahydrofuran (7ml), under nitrogen, was treated dropwise with a solution of sodium bis(trimethylsilyl)amide (1M, 4.4ml, 4.4mmol). After stirring for a further 15 minutes the mixture was treated with a solution of di-*tert*-butyldicarboxylate (440 mg, 2mmol) in anhydrous tetrahydrofuran (3ml). The resulting thick slurry was stirred at ambient temperature for 18 hours then partitioned between ethyl acetate (3 lots of 50ml) and water (50ml). The combined organics were dried over sodium sulfate then concentrated under vacuum. The residue was subjected to chromatography on silica gel eluting with a mixture of cyclohexane and ethyl acetate (9:1, v/v) to give 3-iodo-6-(*tert*-butoxycarbonylamino)pyridine as a white solid (0.56g). ^{1}H NMR ($CDCl_3$, 500MHz) δ8.50 (s, 1H), 7.92 (m, 1H), 7.85 (m, 1H), 1.58 (s, 9H). MS(EI): 319(M^+- H).

B. *N,N*-Bis-(*tert*-butoxycarbonyl)-3-{1-[3-(6-*tert*-butoxycarbonylamino-pyridin-3-yl)ethynyl-benzoyl]-piperidin-4-yl}-benzylamine

By proceeding in a similar manner to the method described in EXAMPLE 5B, but using 3-iodo-6-(*tert*-butoxycarbonylamino)pyridine in place of iodobenzene, there was prepared *N,N*-bis-(*tert*-butoxycarbonyl)-3-{1-[3-(6-*tert*-butoxycarbonylamino-pyridin-3-yl)ethynyl-benzoyl]-piperidin-4-yl}-benzylamine as a yellow amorphous solid. ^{1}H NMR ($CDCl_3$, 500MHz): δ 8.41 (s, 1H), 7.99 (m, 1H), 7.80 (m, 1H), 7.61 (s, 1H), 7.59 (m, 2H), 7.40 (m, 2H), 7.23 (m, 1H), 7.12 (m, 3H), 4.90 (br m,1H), 4.79 (s, 2H), 3.84 (br m, 1H), 3.15 (br m, 1H), 2.84 (br m, 1H), 2.78 (br m, 1H), 1.95 (m, 1H), 1.80 (m, 3H), 1.55 (s, 9H), 1.42 (s, 18H). MS(EI): 710(M^+).

C. 3-{1-[3-(6-Amino-pyridin-3-yl)ethynyl-benzoyl]-piperidin-4-yl}-benzylamine hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 5C but using *N,N*-bis-(*tert*-butoxycarbonyl)-3-{1-[3-(6-*tert*-butoxycarbonylamino -pyridin-3-yl)ethynyl-benzoyl]-piperidin-4-yl}-benzylamine, there was prepared 3-{1-[3-(6-Amino-pyridin-3-yl)ethynyl-benzoyl]-piperidin-4-yl}-benzylamine hydrochloride as a white amorphous solid.). ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.22 (s, 1H), 8.15 (br s, 2H), 7.90 (m, 1H), 7.60-7.41 (m, 5H), 7.30 (m, 3H), 6.93 (m, 1H), 4.60 (br m, 1H), 4.00 (m, 2H), 3.62 (br m, 1H), 3.25 (br m, 1H), 2.85 (br m, 2H), 1.84 (br m, 1H), 1.60 (br m, 3H). MS(EI): 411(M^++H).

EXAMPLE 8

3-{1-[3-(4-hydroxymethylphenyl)ethynyl-benzoyl]-piperidin-4-yl}-benzylamine hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 5, but using 4-iodobenzylalcohol (prepared according to the procedure described by D.S.Tan et al., J. Am. Chem. Soc., 1998, 120(33), pages 8565-8566) in place of iodobenzene, there was prepared the title compound as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 7.61-7.25 (m, 12H), 5.33 (m, 1H), 4.62 (br m, 1H), 4.58 (m, 2H), 4.00 (s, 2H), 3.62 (br m, 1H), 3.20 (br m, 1H), 2.83 (br m, 2H), 1.84 (br m, 1H), 1.72-1.62 (br m, 3H). MS(EI): 425(M^++H).

EXAMPLE 9

3-[1-(3-Phenylethyl-benzoyl)-piperidin-4-yl]-benzylamine hydrochloride

A solution of *N,N*-bis-(*tert*-butoxycarbonyl)-3-[1-(3-phenylethynyl-benzoyl)-piperidin-4-yl]-benzylamine (110mg, 0.18mmol, EXAMPLE 5B) in ethanol (10ml) was treated with 10% palladium on carbon (20mg) and then stirred at ambient temperature under an atmosphere of hydrogen for 8 hours. The reaction mixture was filtered through a short pad of hyflo and then concentrated under vacuum to give a white amorphous solid (0.54g). This material was treated with methanolic hydrogen chloride according to the procedure described in EXAMPLE 5C to give the title compound as white amorphous solid (25mg). ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 7.40-7.15 (m, 13H), 4.62 (br m, 1H), 4.00 (s, 2H), 3.60 (br m, 1H), 3.10 (br m, 1H), 2.80 (br m, 2H), 1.85 (br m, 1H), 1.60 (br m, 3H), MS(EI): 399(M^++H).

EXAMPLE 10

3-{1-[3-(4-hydroxyphenyl)ethyl-benzoyl]-piperidin-4-yl}-benzylamine hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 9, but using *N,N*-bis-(*tert*-butoxycarbonyl)-3-{1-[3-(4-hydroxyphenyl)ethynyl-benzoyl]-piperidin-4-yl}-benzylamine (EXAMPLE 6A), there was prepared the title compound as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 9.18 (br s, 1H), 7.41 (s, 1H), 7.35 (m, 5H), 7.20 (m, 2H), 6.98 (m, 2H), 6.60 (m, 2H), 4.82 (br m,1H), 4.00 (m, 2H), 3.80 (br m, 1H), 3.10 (br m, 1H), 2.80 (m, 6H), 1.82 (br m, 1H), 1.70 (br m, 1H), 1.60 (br m, 2H). MS(EI): 415(M^++ H).

EXAMPLE 11

3-{1-[3-(6-amino-pyridin-3-yl)ethyl-benzoyl]-piperidin-4-yl}-benzylamine hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 9, but using *N,N*-bis-(*tert*-butoxycarbonyl)-3-{1-[3-(6-*tert*-butoxycarbonylamino-pyridin-3-yl)ethynyl-benzoyl]-

piperidin-4-yl}-benzylamine (EXAMPLE 7B), there was prepared the title compound as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 7.90 (br s, 2H), 7.85 (m, 1H), 7.76 (s, 1H), 7.43 (s, 1H), 7.40-7.30 (m, 5H), 7.24 (m, 2H), 6.93 (m, 1H), 4.61 (br m, 1H), 4.00 (m, 2H), 3.60 (br m, 1H), 3.15 (br m, 1H), 2.92 (br m, 6H), 1.90 (br m, 1H), 1.70 (br m, 1H), 1.60 (br m, 2H). MS(EI): 415(M^++H).

EXAMPLE 12

3-[1-(4-Phenylethyl-thiophene-2-carbonyl)-piperidin-4-yl]-benzylamine hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 1, but using 4-phenylethyl-thiophene-2-carboxylic acid (prepared according the procedure described by S.Gronowitz et al., Heterocycles, 1981, 15(2), pages 947-959) in place of 5-phenylethynyl-pyridine-3-carboxylic acid, there was prepared the title compound as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 7.41-7.18 (m, 11H), 4.37 (br m, 1H), 4.00 (m, 2H), 3.05 (br m, 2H), 2.98 (s, 4H), 2.85 (br m, 2H), 1.90 (br m, 2H), 1.60 (br m, 2H), MS(EI): 405(M^++H).

EXAMPLE 13

3-[1-(5-Phenylethyl-thiophene-2-carbonyl)-piperidin-4-yl]-benzylamine hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 2, but using 5-phenylethynyl-thiophene-2-carboxylic acid in place of 5-phenylethynyl-pyridine-3-carboxylic acid, there was prepared the title compound as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 7.41 (s, 1H), 7.38-7.20 (m, 9H), 6.83 (s, 1H), 4.40 (br m, 1H), 4.00 (br s, 2H), 3.12 (m, 2H), 3.08 (br m, 2H), 2.98 (m, 2H), 2.85 (br m, 2H), 1.82 (br m, 2H), 1.60 (br m, 2H), MS(EI): 405(M^++H).

EXAMPLE 14

3-{1-[3-(Benzooxazo-2-yl)-benzoyl]-piperidin-4-yl}-benzylamine hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 1, but using 3-(benzooxazo-2-yl)-benzoic acid (prepared according to the procedure described by V.F.Bystrov et al., Zh. Obshch. Khim., 1968, 38(5), pages 1001-1005) in place of 5-phenylethynyl-pyridine-3-carboxylic acid, there was prepared the title compound as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.28 (m, 1H), 8.19 (s, 1H), 7.81 (m, 2H), 7.50 (m, 2H), 7.43 (m, 3H), 7.30 (m, 3H), 4.66 (br m, 1H), 4.00 (m, 2H), 3.70 (br m, 1H), 3.25 (br m, 1H), 2.92 (br m, 1H), 2.82 (br m, 1H), 1.90 (br m, 1H), 1.70 (br m, 3H), MS(EI): 412(M^++H).

EXAMPLE 15

3-[1-(3-Phenoxymethyl-benzoyl)-piperidin-4-yl]-benzylamine hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 1, but using 3-phenoxymethyl-benzoic acid (prepared according to the procedure of H.Oelschlaeger et al., Arch. Pharm. (Weinheim, Ger.), 1978, 311(2), pages 81-97) in place of 5-phenylethynyl-pyridine-3-carboxylic acid, there was prepared the title compound as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 7.58-7.25 (m, 10H), 7.00 (m, 2H), 6.96 (m, 1H), 5.18 (s, 2H), 4.62 (br m, 1H), 4.00 (m, 2H), 3.62 (br m, 1H), 3.18 (br m, 1H), 2.82 (br m, 2H), 1.88 (br m, 1H), 1.65 (br m, 3H), MS(EI): 401(M^++H).

EXAMPLE 16

3-{1-[3-(2-*E*-Phenylethenyl)-benzoyl]-piperidin-4-yl}-benzylamine hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 1, but using 3-(2-*E*-phenylethenyl)-benzoic acid (prepared according to the procedure of N.A.Bumagin et al., Zh. Org. Khim., 1995, 31(4), pages481-487) in place of 5-phenylethynyl-pyridine-3-carboxylic acid, there was prepared the title compound as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 7.71 (m, 1H), 7.63 (m, 3H), 7.50 (m, 2H), 7.40-7.25 (m, 9H), 4.63 (br m, 1H), 4.00 (s, 2H), 3.71 (br m, 1H), 3.10 (br m, 1H), 2.84 (br m, 2H), 1.88 (br m, 1H), 1.70 (br m, 3H), MS(EI): 397(M^++H).

EXAMPLE 17

4-Fluoro-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride

A. 4-(Pinacolatoboronyl)-1,2,3,6-tetrahydro-pyridine trifluoroacetate

A solution of lithium diisopropylamine (59mmol) in anhydrous tetrahydrofuran (50ml) at -78°C was treated dropwise with a solution of *tert*-butyl 4-oxo-1-piperidinecarboxylate (10.7g, 54mmol) in anhydrous tetrahydrofuran (70ml). After stirring at -78°C for a further 20 minutes the reaction mixture was treated with a solution of N-phenyltrifluoromethanesulfonimide (21.2g, 59mmol) in anhydrous tetrahydrofuran (90ml). The resultant orange suspension was warmed to 0°C, then stirred at 0°C for 3 hours and then concentrated under vacuum. The residue was subjected to chromatography on silica gel eluting with a mixture of pentane and dichloromethane (1:1, v/v) and then subjected to chromatography on alumina eluting with a mixture of pentane and ethyl acetate (9:1, v/v) to yield *tert*-butyl 1,2,3,6-tetrahydro-4-(trifluoromethylsulfonyloxy)-pyridine-1-carboxylate as a yellow oil (15g). A portion of this

material (1.72g, 5.2mmol) was dissolved in anhydrous dioxane (30ml) and the solution was treated with bis(pinacolato)diboron (1.46g, 5.75mmol), potassium acetate (1.54g, 15.7mmol), (diphenylphosphino)-ferrocene (86mg, 0.16mmol) and [1,1'-bis-(diphenylphosphino)ferroceno]-dichloropalladium (II) (114mg, 0.16mmol). The reaction mixture was heated at 80°C under an atmosphere of nitrogen for 18 hours, then cooled and then concentrated under vacuum. The residue was partitioned between ethyl acetate (2 lots of 100ml) and water (100ml). The combined organic extracts were dried over magnesium sulfate then concentrated under vacuum. The resultant oil was subjected to chromatography on silica gel eluting with a mixture of ethyl acetate and pentane (1:8, v/v) to yield a yellow oil (1.4g). A solution of this material in dichloromethane (10ml), cooled to 0°C, was treated with trifluoracetic acid (3.9ml). The mixture was stirred at ambient temperature for 2 hours then concentrated under vacuum to leave 4-(pinacolatoboronyl)-1,2,3,6-tetrahydro-pyridine trifluoroacetate as a brown oil. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.76 (br s, 2H), 6.39 (br s, 1H), 3.61 (br m, 2H), 3.11 (br m, 2H), 2.23 (br m, 2H), 1.20 (s 12H). MS(EI): 210(M^++ H).

B. 1-(5-Phenylethyl-pyridine-3-carbonyl)-4-(pinacolatoboronyl)-1,2,3,6-tetrahydro-pyridine

A solution of 5-phenylethyl-pyridine-3-carboxylic acid (0.46g, 2.0mmol, EXAMPLE 2A) in anhydrous dimethylformamide (9ml) was treated with O-(7-azabenzotriazol-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate (0.87g, 2.2mmol) and diisopropylethylamine (1.7ml, 10mmol). This mixture was stirred at ambient temperature for 10 minutes then treated with a solution of 4-(pinacolatoboronyl)-1,2,3,6-tetrahydro-pyridine trifluoroacetate (0.81g, 2.5mmol) in dimethylformamide (9ml). The reaction mixture was stirred at ambient temperature for 18 hours and then concentrated under vacuum. The residue was partitioned between dichloromethane (2 lots of 50ml) and saturated aqueous sodium bicarbonate (15ml). The combined organic layers were dried over magnesium sulfate and then concentrated under vacuum. The residue was subjected to chromatography on silica gel eluting with ethyl acetate to give 1-(5-phenylethyl-pyridine-3-carbonyl)-4-(pinacolatoboronyl)-1,2,3,6-tetrahydro-pyridine as a brown amorphous solid (0.73g). ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.49 (s, 1H), 8.40 (s, 1H), 7.61 (s, 1H), 7.20 (m, 5H), 6.48 (br s, 1H), 4.18 (br m, 1H), 3.83 (br m, 1H), 3.62 (br m, 1H), 3.23 (br m, 1H), 2.98 (m, 4H), 2.17 (br m, 2H), 1.20 (s, 12H). MS(EI): 441(M^++Na).

C. N-(*tert*-Butoxycarbonyl)-3-bromo-4-fluoro-benzylamine

A mixture of 3-bromo-4 fluoro-benzylamine hydrochloride (2.41g, 10mmol), triethylamine (2.8ml, 20mmol), and di-*tert*-butoxycarbonate (1.8g, 10.3mmol) in dichloromethane (20ml) was stirred at ambient temperature for 18 hours then washed with water (20ml). The organic phase

was dried over magnesium sulfate and then concentrated under vacuum. The residue was subjected to chromatography on silica gel eluting with a mixture of cyclohexane and ethyl acetate (3:1, v/v) to give N-(*tert*-butoxycarbonyl)-3-bromo-4-fluoro-benzylamine as a white solid (1.4g). ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.45 (m, 1H), 7.20 (m, 1H), 7.08 (m, 1H), 4.85 (br s, 1H), 4.21 (m, 2H), 1.42 (s, 9H).

D. *N*-(*tert*-Butoxycarbonyl)-4-fluoro-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-1,2,3,6-tetrahydro-pyridin-4-yl]-benzylamine

A mixture of N-(*tert*-butoxycarbonyl)-3-bromo-4-fluoro-benzylamine (0.28g, 0.92mmol), anhydrous dimethylformamide (10ml), 1-(5-phenylethyl-pyridine-3-carbonyl)-4-(pinacolatoboronyl)-1,2,3,6-tetrahydro-pyridine (0.37g, 0.88mmol), potassium carbonate (0.36g, 2.6mmol) and [1,1'-bis-(diphenylphosphino)ferroceno]dichloropalladium (II)-dichloromethane complex (43 mg, 0.05mmol) was heated at 80°C under an atmosphere of nitrogen for 18 hours. The reaction mixture was cooled to room temperature and then concentrated under vacuum. The residue was partitioned between ethyl acetate (2 lots of 50ml) and water (10ml). The combined organic extracts were dried over magnesium sulfate and then concentrated under vacuum. The resultant oil was subjected to chromatography on silica gel eluting with ethyl acetate to yield *N*-(*tert*-butoxycarbonyl)-4-fluoro-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-1,2,3,6-tetrahydro-pyridin-4-yl]-benzylamine as a pale yellow oil (0.18g). ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.58 (s, 1H), 8.50 (s, 1H), 7.51 (s, 1H), 7.25 (m, 2H), 7.18 (m, 5H), 7.00 (m, 1H), 6.01 (br s, 1H), 4.83 (br s, 1H), 4.38 (br m, 1H), 4.27 (m, 2H), 3.98 (br m, 2H), 3.50 (br m, 1H), 2.99 (m, 4H), 2.60 (br m, 1H), 2.50 (br m, 1H). MS(EI): 538(M^++ Na).

E. 4-Fluoro-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride

A solution of *N*-(*tert*-butoxycarbonyl)-4-fluoro-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-1,2,3,6-tetrahydro-pyridin-4-yl]-benzylamine in ethanol (12ml) was treated with 10% palladium on carbon (75mg) and the mixture was stirred at ambient temperature under an atmosphere of hydrogen for 72 hours. The reaction mixture was filtered through a short pad of hyflo and the filtrate was concentrated under vacuum to give *N*-(*tert*-butoxycarbonyl-4-fluoro-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine as colourless oil (85mg). This material was dissolved in methanol (10ml) and the solution was cooled to 0°C and then saturated with hydrogen chloride gas. This mixture was stirred at ambient temperature for 4 hours and then concentrated to dryness under vacuum. The residue was triturated with a mixture of dichloromethane and diethyl ether to give 4-fluoro-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-

piperidin-4-yl]-benzylamine di-hydrochloride as a white amorphous solid (50mg). ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.60 (s, 1H), 8.58 (s, 1H), 7.83 (s, 1H), 7.60 (m, 1H), 7.38 (m, 1H), 7.20 (m, 6H), 4.61 (br m, 1H), 4.00 (m, 2H), 3.50 (br m, 1H), 3.20 (br m, 2H), 2.99 (m, 2H), 2.95 (m, 2H), 2.90 (br m, 1H), 1.82 (br m, 1H), 1.65 (br m, 3H). MS(EI): 418(M^++H).

EXAMPLE 18

4-Methyl-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride

A. 4-[*N,N*-Bis-(*tert*-butoxycarbonyl)aminomethyl]-2-bromo-toluene

A solution of 4-methyl-3-bromobenzylbromide (1.63g, 6.2mmol, prepared according to the procedure described in International Patent Application No. WO 0009475) and di-*tert*-butyliminodicarboxylate (1.48g, 6.8mmol) in anhydrous tetrahydrofuran (15ml) was treated portionwise with sodium hydride (0.27g of 60% dispersion in mineral oil, 6.8mmol). The mixture was stirred at ambient temperature for 18 hours then partitioned between saturated aqueous ammonium chloride solution (20ml) and ethyl acetate (3 lots of 80ml). The combined organic layers were washed with brine (80ml), then dried over magnesium sulfate and concentrated under vacuum. The residue was subjected to chromatography on silica gel eluting with a mixture of cyclohexane and diethyl ether (9:1, v/v) to give 4-[*N,N*-bis-(*tert*-butoxycarbonyl)aminomethyl]-2-bromo-toluene as a pale yellow oil (2.4g). ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.45 (s, 1H), 7.18 (m, 2H), 4.71 (s, 2H), 2.38 (s, 3H), 1.43 (s, 18H).

B. 4-Methyl-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 17, but using 4-[*N,N*-bis-(*tert*-butoxycarbonyl)aminomethyl]-2-bromo-toluene in place of N-(*tert*-butoxycarbonyl)-3-bromo-4-fluoro-benzylamine, there was prepared 4-methyl-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride as a white amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.60 (m, 2H), 7.83 (s, 1H), 7.41 (s, 1H), 7.20 (m, 7H), 4.62 (br m, 1H), 3.98 (m, 2H), 3.45 (br m, 1H), 3.20 (br m, 1H), 3.07 (m, 2H), 3.00 (m, 2H), 2.95 (m, 2H), 2.32 (s, 3H), 1.80 (br m, 1H), 1.70 (br m, 3H). MS(EI): 414(M^++H).

EXAMPLE 19

3-{1-[3-(5-Phenyl-1,3,4-oxadiazol-2-yl)-phenylcarbonyl]-piperidin-4-yl}-benzylamine hydrochloride

A. 3-(5-Phenyl-1,3,4-oxadiazol-2-yl)-benzoic acid

A mixture of methyl hydrogen isopthalalate (1.8g, 10mmol), benzoic hydrazide (1.4g, 10mmol) and phosphorous oxychloride (20ml), under an atmosphere of nitrogen, was heated at 120°C for 18 hours, then cooled to room temperature and then poured into ice water (500ml). This mixture was treated with solid sodium carbonate until the aqueous layer was basic (pH 8 -9) and the resultant pink solid was filtered. This material was treated with 100ml methanol and the suspension was treated with sodium hydroxide solution (30ml, 1M). The reaction mixture was heated at reflux for 4 hours, then cooled and then concentrated to dryness. The residue was dissolved in water (100ml) and the solution was acidified to pH 3 by addition of concentrated hydrochloric acid. The resultant precipitate was filtered, then dried and then subjected to chromatography on silica gel eluting with a mixture of dichloromethane and methanol (98:2, v/v) to yield 3-(5-phenyl-1,3,4-oxadiazol-2-yl)-benzoic acid as a white solid (600mg). ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.80 (s, 1H), 8.38 (m, 1H), 8.18 (m, 3H), 7.78 (m, 1H), 7.62 (m, 3H). MS(EI): 265(M^+- H).

B. 3-{1-[3-(5-Phenyl-1,3,4-oxadiazol-2-yl)-phenylcarbonyl]-piperidin-4-yl}-benzylamine hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 1, but using 3-(5-phenyl-1,3,4-oxadiazol-2-yl)-benzoic acid in place of 5-phenylethynyl-pyridine-3-carboxylic acid, there was prepared 3-{1-[3-(5-phenyl-1,3,4-oxadiazol-2-yl)-phenylcarbonyl]-piperidin-4-yl} benzylamine hydrochloride as a pale yellow amorphous solid. ^{1}H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.21 (m, 1H), 8.20 (m, 3H), 7.75 (m, 2H), 7.68 (m, 3H), 7.50 (s, 1H), 7.37 (m, 3H), 4.70 (br m, 1H), 4.00 (m, 2H), 3.70 (br m, 1H), 3.25 (br m, 1H), 2.90 (br m, 1H), 2.85 (br m, 1H), 1.88 (br m, 1H), 1.70 (m, 3H), MS(EI): 439(M^++H).

EXAMPLE 20

3-[1-(Indole-6-carbonyl)-piperidin-4-yl]-benzylamine trifluoroacetate

A solution of diisopropylamine in dimethylformamide (1ml, 180μM) in a glass vial was treated with a solution of O-(7-azabenzotriazol-1-yl)-1,1,3,3-tetramethyluronium hexafluorophosphate in dimethylformamide (1ml, 60μM) followed by a solution of indole-6-carboxylic acid in dimethylformamide (1ml, 60μM). After standing at ambient temperature for 15 minutes the

mixture was treated with a solution of 4-{3-[*N*,*N*-bis-(*tert*-butoxycarbonyl)aminomethyl]-phenyl}-piperidine in dimethylformamide(1ml, 60μM, EXAMPLE 1B). The reaction mixture was allowed to stand at ambient temperature for 18 hours then evaporated. The residue was treated with chloroform (5ml) and aqueous sodium carbonate solution (5%). This mixture was shaken gently for 30 minutes, poured into a fritted polypropylene tube and the organic layer which passes through the frit collected in a glass vial. The chloroform was evaporated under vacuum and the residue was treated with a mixture of trifluoroacetic acid, dichloromethane and water (4ml, 55/40/5, v/v/v). This mixture was shaken gently for 2 hours and then evaporated to leave the title compound as a yellow oil. LC-MS: R_T = 3.43 minutes (>96% by ELSD); MS (ES^+), 334 (MH^+).

EXAMPLE 21

3-[1-(Coumarin-3-carbonyl)-piperidin-4-yl]-benzylamine trifluoroacetate

By proceeding in a similar manner to the method described in EXAMPLE 20, but using coumarin-3-carboxylic acid in place of indole-6-carboxylic acid, there was prepared the title compound as a yellow oil. LC-MS: R_T = 3.15 minutes (>86% by ELSD); MS (ES^+), 363 (MH^+).

EXAMPLE 22

3-[1-(Naphthyl-2-carbonyl)-piperidin-4-yl]-benzylamine trifluoroacetate

By proceeding in a similar manner to the method described in EXAMPLE 20, but using 2-naphthoic acid in place of indole-6-carboxylic acid, there was prepared the title compound as a yellow oil. LC-MS: R_T = 3.66 minutes (100% by ELSD); MS (ES^+), 345 (MH^+).

EXAMPLE 23

3-{1-[3-(2-Naphthylthio)propionyl]-piperidin-4-yl}-benzylamine trifluoroacetate

By proceeding in a similar manner to the method described in EXAMPLE 20, but using 3-(2-naphthylthio)propionic acid in place of indole-6-carboxylic acid, there was prepared the title compound as a yellow oil. LC-MS: R_T = 4.00minutes (>95% by ELSD); MS (ES^+), 405(MH^+).

EXAMPLE 24

3-{1-[4-(Indol-3-yl)butanoyl]-piperidin-4-yl}-benzylamine trifluoroacetate

By proceeding in a similar manner to the method described in EXAMPLE 20, but using 4-(indol-3-yl)butanoic acid in place of indole-6-carboxylic acid, there was prepared the title compound as a yellow oil. LC-MS: R_T = 3.64 minutes (>90% by ELSD); MS (ES^+), 376(MH^+).

EXAMPLE 25

3-{1-[4-(4-Biphenyl)-4-ketobutanoyl]-piperidin-4-yl}-benzylamine trifluoroacetate

By proceeding in a similar manner to the method described in EXAMPLE 20, but using 4-(4-biphenyl)-4-ketobutanoic acid in place of indole-6-carboxylic acid, there was prepared the title compound as a yellow oil. LC-MS: R_T = 4.00 minutes (100% by ELSD); MS (ES^+), 427(MH^+).

EXAMPLE 26

3-[1-(3-Benzyloxybenzoyl)-piperidin-4-yl]-benzylamine trifluoroacetate

By proceeding in a similar manner to the method described in EXAMPLE 23, but using 3-benzyloxybenzoic acid in place of indole-6-carboxylic acid, there was prepared the title compound as a yellow oil. LC-MS: R_T = 3.87 minutes (100% by ELSD); MS (ES^+), 401 (MH^+).

EXAMPLE 27

3-[1-(5-Phenylethynyl-thiophene-2-carbonyl)-piperidin-4-yl]-benzylamine trifluoroacetate

By proceeding in a similar manner to the method described in EXAMPLE 20, but using 5-phenylethynyl-thiophene-2-carboxylic acid in place of indole-6-carboxylic acid, there was prepared the title compound as a yellow oil. LC-MS: R_T = 4.09 minutes (>97% by ELSD); MS(ES^+), 410(MH^+).

EXAMPLE 28

3-[1-(4-Phenylethynyl-thiophene-2-carbonyl)-piperidin-4-yl]-benzylamine trifluoroacetate

By proceeding in a similar manner to the method described in EXAMPLE 20, but using 4-phenylethynyl-thiophene-2-carboxylic acid in place of indole-6-carboxylic acid, there was prepared the title compound as a yellow oil. LC-MS: R_T = 4.08 minutes (>96% by ELSD); MS(ES^+), 401(MH^+).

EXAMPLE 29

3-(1-Benzoyl-piperidin-4-yl)-benzylamine trifluoroacetate

By proceeding in a similar manner to the method described in EXAMPLE 20, but using benzoic acid in place of indole-6-carboxylic acid, there was prepared the title compound as a yellow oil. LC-MS: R_T = 3.39 minutes (>95% by ELSD); MS(ES^+) 295(MH^+).

EXAMPLE 30

3-[1-(4-*N*,*N*-Dimethylaminobenzoyl)-piperidin-4-yl]-benzylamine trifluoroacetate

By proceeding in a similar manner to the method described in EXAMPLE 20, but using 4-*N*,*N*-dimethylaminobenzoic acid in place of indole-6-carboxylic acid, there was prepared the title compound as a yellow oil. LC-MS: R_T = 3.32 minutes (100% by ELSD); MS(ES^+) 338(MH^+).

EXAMPLE 31

6-Fluoro-3-[1-(5-phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 17, but using 3-bromo-6-fluoro-benzylamine in place of 3-bromo-4-fluoro-benzylamine, there was prepared the title compound as a pale yellow oil. ^1H NMR [$(CD_3)_2SO$, 500 MHz]: δ 8.44 (m, 2H), 7.61 (s, 1H), 7.40 (m, 1H), 7.20 (m, 6H), 7.05 (m, 1H), 4.62 (br m, 1H), 3.98 (m, 2H), 3.45 (br m, 1H), 3.20 (br m, 1H), 3.07 (m, 2H), 3.00 (m, 2H), 2.95 (m, 2H) 1.80 (br m, 1H), 1.70 (br m, 3H). MS(EI): 418(M^++H).

EXAMPLE 32

1-{3-[1-(5-Phenylethyl-pyridine-3-carbonyl)-piperidin-4-yl]-phenyl}ethylamine di-hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 17, but using 1-(3-bromophenyl)ethylamine (prepared according to the procedure of C.P.Chen et al., Tetrahedron Letters, 199, 32(49), pages 7175-7178) in place of 3-bromo-4-fluoro-benzylamine, there was prepared the title compound as a white solid. ^1H NMR[$(CD_3)_2SO$, 500 MHz]: δ 8.62 (m, 2H), 7.95 (s, 1H), 7.50 (s, 1H), 7.20 (m, 8H), 4.62 (br m, 1H), 4.38 (t, J = 6 Hz, 1H), 3.50 (m, 1H), 3.45 (br m, 1H), 3.20 (br m, 1H), 3.07 (m, 2H), 3.00 (m, 2H), 2.95 (m, 2H), 1.87 (br m, 1H), 1.65 (br m, 3H), 1.50 (d, J = 6 Hz, 3H). MS(EI): 414(M^++H).

EXAMPLE 33

3-[1-(4-Hydroxy-quinoline-3-carbonyl)-piperidin-4-yl]-benzylamine

By proceeding in a similar manner to the method described in EXAMPLE 1, but using 4-hydroxy-quinoline-3-carboxylic acid (prepared according to the procedure of K.J.Shah, and E.A.Coats, J. Med. Chem. , 1977, 20(8), pages 1001-1006) in place of 5-phenylethynyl-pyridine-3-carboxylic acid, there was prepared the title compound as a white amorphous solid. ^1H NMR[$(CD_3)_2SO$, 500 MHz]: δ 8.10 (m, 2H), 7.68 (m, 1H), 7.59 (m, 1H), 7.36 (m, 3H), 7.22 (m, 2H), 4.65 (br m, 1H), 4.00 (m, 2H), 3.80 (br m, 1H), 3.35 (br m, 1H), 3.10 (br m, 1H), 3.00 (br m, 1H), 1.90-1.75 (br m, 4H). MS(EI): 362(M^++H).

EXAMPLE 34

3-[1-(6-Phenyl-quinoline-3-carbonyl)-piperidin-4-yl]-benzylamine di-hydrochloride

By proceeding in a similar manner to the method described in EXAMPLE 1, but using 6-phenyl-quinoline-3-carboxylic acid [prepared according to the procedure of J.Biwersi et al., Am. J. Physiol., 1992, 262(1, Pt. 1), C243-C250] in place of 5-phenylethynyl-pyridine-3-carboxylic acid, there was prepared the title compound as a white amorphous solid. ^{1}H NMR[$(CD_3)_2SO$, 500 MHz]: δ 9.00 (s, 1H), 8.59 (s, 1H), 8.41 (s, 1H), 8.10 (m, 2H), 7.85 (m, 2H), 7.58 (m, 2H), 7.45 (m, 2H), 7.34 (m, 3H), 4.75 (br m, 1H), 4.00 (m, 2H), 3.80 (br m, 1H), 3.35 (br m, 1H), 3.00 (br m, 1H), 2.90 (br m, 1H), 1.90-1.75 (br m, 4H). MS(EI): 422(M^++H).

EXAMPLE 35

4-(3-Aminomethyl-phenyl)-1-(5-phenethyl-pyridine-3-carbonyl)-piperidine-4-carbonitrile

A. 3-Cyanobenzylcyanide

A solution of sodium cyanide (9.8g, 200mmole) and tetrabutylammonium bromide (1.61g, 5mmole) in water (50ml) was treated with a solution of a-bromo-m-tolunitrile (19.61g, 100mmole) in dichloromethane (150ml). The mixture was stirred at room temperature for 24 hours then treated with iodomethane (6.2ml, 100mmole) and stirring at room temperature was continued for a further 3 hours. The layers were separated and the organic phase was dried over magnesium sulfate and then evaporated. The residue was triturated with petroleum ether to give 3-cyanobenzylcyanide (14.5g) as a white solid, m.p. 66-69°C. ^{1}H-NMR ($CDCl_3$, 300MHz): δ 3.80-3.90 (s, 2H), 7.49-7.72 (m, 4H).

B. N-tert-Butoxycarbonyl-bis(2-chloroethyl)amine

A mixture of bis(2-chloroethyl)amine hydrochloride (17.85g, 100mmole) and di-tert-butyl dicarbonate (24g, 110mmole) in dichloromethane (100ml)was treated dropwise with a solution of triethylamine (15.3ml, 110mmole) in dichloromethane (50ml) over 30 minutes. After stirring at room temperature for 24 hours the reaction mixture was poured into water. The organic layer was separated, then dried over magnesium sulfate and then evaporated to give N-tert-butoxycarbonyl-bis(2-chloroethyl)amine (24g) as an oil. ^{1}H-NMR ($CDCl_3$, 300MHz): δ 1.37-1.56 (m, 9H), 3.50-3.62 (m, 8H). MS (ES): 242 $(M+H)^+$.

C. 4-(3-Cyanophenyl)-1-tert-butoxycarbonyl-piperidine-4-carbonitrile

A solution 3-cyanobenzylnitrile (2.8g, 19.7mmole)and N-tert-butylcarbonyl-bis(2-chloroethyl)amine (4.77g, 19.7mmole) in anhydrous dimethylformamide (100ml) was treated portionwise with sodium hydride (2.36g, 59.1mmole, 60% dispersion in mineral oil) over 10 minutes. The mixture was stirred at room temperature for 3 days, then poured into water and then extracted with ether. The ether extract was dried over magnesium sulfate, then filtered and then evaporated. The residue was subjected to flash chromatography on silica eluting with a mixture of hexane and ethyl acetate (4:1, v/v) to give 4-(3-cyanophenyl)-1-tert-butoxycarbonyl-piperidine-4-carbonitrileas a white solid: ^{1}H-NMR ($CDCl_3$, 300MHz): δ 1.45(s, 9H), 1.85-2.0(m, 2H), 2.0-2.28(m, 2H), 3.05-3.30(m, 2H), 4.12-4.4(m, 2H), 7.53-7.80(m, 4H).

D. 4-(3-Aminomethyl-phenyl)-1-tert-butoxycarbonyl-piperidine-4-carbonitrile

A mixture 4-(3-cyanophenyl)-1-tert-butoxycarbonyl-piperidine-4-carbonitrile (0.31g, 1mmole) and absolute ethanol (30ml) was treated with concentrated hydrochloric acid (0.08ml, 1mmole) followed by palladium on carbon (30mg, 5%). The mixture was agitated in a Parr shaker for 3 hours at 30 lbs of pressure of hydrogen then filtered through celite. The filtrate was evaporated and the residue was treated with ether. This mixture was stirred for 30 minutes and then filtered. The filter cake was treated with aqueous sodium carbonate and then extracted with dichloromethane to give 4-(3-aminomethyl-phenyl)-1-tert-butoxycarbonyl-piperidine-4-carbonitrile (0.2g). ^{1}H-NMR ($CDCl_3$, 300MHz): δ 1.38-1.60(m, 9H), 1.88-2.18(m, 4H), 3.05-3.30(m, 4H), 3.79-4.0(m, 2H), 7.22-7.85(m, 4H). MS (ES): 316 $(M+H)^+$.

E. 4-(3-Benzyloxycarbonylaminomethyl-phenyl)-1-tert-butoxycarbonyl-piperidine-4-carbonitrile

A solution 4-(3-aminomethyl-phenyl)-1-tert-butoxycarbonyl-piperidine-4-carbonitrile (0.2g) in dichloromethane (15ml) was treated with a few drops of triethylamine followed by a few drops of benzyl chloroformate. After stirring at room temperature for 1 hour the reaction mixture was poured into aqueous sodium carbonate and this mixture was then extracted with dichloromethane. The extract was dried over magnesium sulfate and then evaporated to give 4-(3-benzyloxycarbonylaminomethyl-phenyl)-1-tert-butoxycarbonyl-piperidine-4-carbonitrile which was used directly in the next step without further purification : ^{1}H-NMR ($CDCl_3$, 300MHz): δ 1.39-1.52 (m, 9H), 1.80-2.16(m, 4H), 3.08-3.42(m, 4H), 4.19-4.50(m, 2H), 5.08-5.13(d, 2H), 7.28-7.79(m, 9H). MS (ES) m/e 450 $(M+H)^+$.

F. 4-(3-Benzyloxycarbonylaminomethyl-phenyl)-piperidine-4-carbonitrile

The residue from the previous step was dissolved in dichloromethane containing 1 ml of trifluoroacetic acid and the solution was stirred at room temperature for 45 minutes. The solution was poured into aqueous sodium carbonate and then extracted with dichloromethane. The organic layer was dried and then evaporated. The residue was subjected to flash chromatography on silica eluting with a mixture of hexane and ethyl acetate (7:3, v/v) to give 4-(3-benzyloxycarbonylaminomethyl-phenyl)-piperidine-4-carbonitrile. ^{1}H-NMR ($CDCl_3$, 300MHz): δ 1.80-2.18(m, 4H), 3.07-3.35(m, 4H), 4.28-4.50(m, 2H), 5.09-5.13(d, 2H), 7.10-7.77(m, 9H). MS (ES) m/e 349 $(M+H)^+$.

G. 4-(3-Benzyloxycarbonylaminomethyl-phenyl)-1-(5-phenethyl-pyridine-3-carbonyl)-piperidine-4-carbonitrile

A solution 4-(3-benzyloxycarbonylaminomethyl-phenyl)-piperidine-4-carbonitrile (0.15g, 0.33mmole) in acetonitrile (20ml) was treated with N-ethylmorpholine (0.038g, 0.33mmole) followed by TBTU (0.11g, 0.33mmole). This solution was then treated portionwise with 5-phenethylpyridine-3-carboxylic acid (0.075g, 0.33mmole) over 10 minutes. After stirring at room temperature for 2 hours the reaction mixture was concentrated and the residue was subjected to flash chromatography on silica eluting with a mixture of dichloromethane and methanol (95:5, v/v) to give 4-(3-benzyloxycarbonylaminomethyl-phenyl)-1-(5-phenethyl-pyridine-3-carbonyl)-piperidine-4-carbonitrile (0.2g). ^{1}H-NMR ($CDCl_3$, 300MHz): δ 1.79-2.20 (m, 4H), 2.70-2.82 (m, 4H), 2.90-3.07 (m, 2H), 3.15-3.70 (m, 2H), 4.35-4.40 (d, 2H), 5.07-5.25 (d, 2H), 7.10-7.50 (m, 15H), 8.42-8.58 (m, 2H); MS (ES) m/e 559 $(M+H)^+$.

H. 4-(3-Aminomethyl-phenyl)-1-(5-phenethyl-pyridine-3-carbonyl)-piperidine-4-carbonitrile trifluoroacetate

A solution 4-(3-benzyloxycarbonylaminomethyl-phenyl)-1-(5-phenethyl-pyridine-3-carbonyl)-piperidine-4-carbonitrile (0.15g) in glacial acetic acid (5ml) was treated with hydrogen bromide in acetic acid (1ml, 30%). After stirring at room temperature for 2 hours the solution was poured into a saturated sodium carbonate solution and the mixture was extracted with dichloromethane. The extract was dried over magnesium sulfate and then evaporated. The residue was subjected to Rainin HPLC using 10-100% (acetonitrile-0.1% aqueous TFA) to yield 4-(3-aminomethyl-phenyl)-1-(5-phenethyl-pyridine-3-carbonyl)-piperidine-4-carbonitrile trifluoroacetate (16mg) isolated as a white solid the trifluoroacetic acid salt. ^{1}H-NMR [$(CD_3)_2SO$, 300MHz): δ 1.95-2.30 (m, 4H), 2.45-2.62(m, 2H), 2.88-3.12(m, 4H), 3.70-4.20 (m, 2H), 7.12-7.38(m, 4H), 7.45-7.82(m, 6H), 8.50-8.65(m, 2H). MS (ES) m/e 425 $(M+H)^+$.

EXAMPLE 36

[4-(3-Aminomethylphenyl)piperidin-1-yl]-(3,4-dichlorophenyl)methanone trifluoroacetate

TFP resin (125mg of 1.25mmol/g resin, with 100% loading of acid, 156mmol, prepared according to the procedure described by J.M.Salvino et. al. in International Patent Application Publication No. WO 99/67228) was swollen in dichloromethane (2.5mL) for 15 minutes then treated with a solution of 4-[3-(N,N-di-tert-butoxycarbonylaminomethyl)phenyl]piperidine (40mg, 100mmol) in dichloromethane (2.5mL). The mixture was sealed in the reaction vessel then left shaking for 8 hours. The resin was filtered, then washed with dichloromethane (2mL) [TLC (5% MeOH / EtOAc) showed single product spot (no baseline amine)] and then treated with trifluoroacetic acid (0.5mL). After shaking for 2 hours TLC showed no residual intermediate and the reaction mixture was evaporated. The residue [97% purity by HPLC: R_T = 7.32 minutes; 10 micron C_{18} reverse phase column (4.6mm x 10cm) eluting with 10-100% acetonitrile and water containing 0.1%trifluoroacetic acid] was dissolved in water (50mL) and the solution was lyophilized to give the title compound as an amorphous white solid. ^{1}H NMR [$(CD_3)_2SO$, 300MHz]: δ 8.15 (br s, 3H), 7.72-7.69 (m, 2H), 7.40 (dd, 1H), 7.36-7.25 (m, 4H), 4.66-4.51 (m, 1H), 4.05-3.96 (m, 2H), 3.69-3.48 (m, 1H), 3.30-3.11 (m, 1H), 2.91-2.73 (m, 2H), 1.90-1.54 (m, 4H). MS(Ion spray): 363 and 365(M^++1).

IN VITRO TEST PROCEDURE

Tryptase Inhibition Activity:

Tryptase inhibition activity is confirmed using either isolated human lung tryptase or recombinant human β tryptase expressed in yeast cells. Essentially equivalent results are obtained using isolated native enzyme or the expressed enzyme. The assay procedure employs a 96 well microplate (Costar 3590) using L-pyroglutamyl-L-prolyl-L-arginine-*para*-nitroanilide (S2366: Quadratech) as substrate (essentially as described by McEuen *et. al.* Biochem Pharm, 1996, 52, pages 331-340). Assays are performed at room temperature using 0.5mM substrate (2 x K_m) and the microplate is read on a microplate reader (Beckman Biomek Plate reader) at 405nm wavelength.. To determine the compound concentration that inhibits half of the enzyme activity (IC_{50}), the fraction of control activity (FCA) is plotted as a function of the inhibitor concentration (I).